QUALITY ASSURANCE PROJECT PLAN ADDENDUM

Radiologically Impacted Material in Areas 1 and 2
West Lake Landfill Operable Unit -1
Bridgeton, Missouri

October, 2015
Prepared by S.S. Papadopulos & Associates, Inc.

Revision 1.1

10/21/15

Matthew Tonkin PhD Date
S.S. Papadopulos & Associates, Inc.
Project Manager

10/21/15

Erica DiFilippo, PhD Date
S.S. Papadopulos & Associates, Inc.
Quality Assurance Officer

10/21/15

Ohannes Sivaslian, PE Date
S.S. Papadopulos & Associates, Inc.
Corporate Health & Safety Officer

QUALITY ASSURANCE PROJECT PLAN ADDENDUM

Radiologically Impacted Material in Areas 1 and 2

West Lake Landfill Operable Unit -1

Bridgeton, Missouri

October 2015 – Revision 1.1

DISTRIBUTION LIST

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address
Matthew Tonkin	Project Manager	S.S. Papadopulos & Associates, Inc. (SSP&A)	(301) 718-8900	(301) 718-8909	hcohen@sspa.com
Erica DiFilippo	Quality Assurance Manager	S.S. Papadopulos & Associates, Inc. (SSP&A)	(301) 500-2260	(301) 718-8909	ericad@sspa.com
Ohannes Sivaslian	Corporate Health & Safety Officer	S.S. Papadopulos & Associates, Inc. (SSP&A)	(301) 718-8900	(301) 718-8909	os@sspa.com
Paul V. Rosasco	Project Coordinator	Engineering Management Support, Inc. (EMSI)	(303)-940-3426	-	paulrosasco@emsidenver.com
Dan Feezor	dfeezor@feezorengineering.com	Feezor Engineering, Inc. (FE)	217-483-3118	-	dfeezor@feezorengineering.com
Bill Abernathy	Residuals Manager/Project Geologist	Feezor Engineering, Inc. (FE)	314-502-1299	-	babernathy@feezorengineering.com
Bradley Vann	Remedial Project Manager	U.S. Environmental Protection Agency	913-551-7611	913-551-9611	Vann.Bradley@epa.gov
Barry Stephenson	President/CEO	Materials & Chemistry Laboratory, Inc. (MCL)	865-576-0201	-	bstephenson@mcl-inc.com
Michael R. McDougall	Laboratory Manager	Eberline Analytical	865-481-0683 (x128)	-	mike.mcdougall@eberlineservices.com

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address

Table of Contents

Page

List of Tables

Attachments

List of Tables

Attachments

List of Acronyms

% R - Percent Recovery
CEC - Cation-Exchange-Capacity
CSWP - Core Sampling (Phase 1B, 1C, and 2) Work Plan – Revision 1
DQO – Data Quality Objections
EMSI - Engineering Management Support Inc.
EPA - United States Environmental Protection Agency
FE – Feezor Engineering
g – grams
IDL – Instrument Detection Limit
kg - kilograms
L- Liters
LCS - Laboratory Control Samples
MCL – Materials & Chemistry Laboratory, Inc.
MDL – Method Detection Limit
meq - milliequivalents
mg – milligrams
MS – Matrix Spike
MSD – Matrix Spike Duplicate
MSW - Municipal Solid Waste
PARCCS - Precision, Accuracy, Representativeness, Comparability, Completeness, & Sensitivity
pCi – picocuires
QAPP – Quality Assurance Project Plan
QA/QC - Quality Assurance/Quality Control
RIM – Radiologically Impacted Material
RL – Reporting Limit
RPD – Relative Percent Difference
SBLT - Sequential Batch Leaching Tests
SEM/EDS - Scanning Electron Microscope with Energy Dispersive X-ray Spectrometry
SPLP - Synthetic Precipitation Leaching Procedure
SSP&A - S.S Papadopulos & Associates, Inc.
TCLP - Toxic Characteristic Leaching Potential
TOC – Total Organic Carbon
wt% - Percentage by weight
XRD – X-ray diffraction

Section 1
Project/Task Organization

The organization and management of the project is detailed in the January 8, 2014 Core Sampling (Phase 1B, 1C, and 2) Work Plan – Revision 1 (CSWP) prepared by Feezor Engineering., Inc. (FE). S.S. Papadopulos & Associates, Inc. (SSP&A) has been retained to assist Engineering Management Support Inc. (EMSI) in the fate and transport evaluations at the site as requested by the United States Environmental Protection Agency (EPA).

Section 2
Problem Definition/Background

West Lake Landfill contains, in places, radiologically impacted material that is referred to as RIM. Areas 1 and 2 of West Lake Landfill are identified by the EPA as Operable Unit 1 of the West Lake Landfill Superfund Site. Remedial actions to address RIM are being directed by EPA.

2.1 Project/Task Description

As part of the remedial actions directed by the EPA to address RIM, EMSI submitted a work plan (Work Plan) for additional characterization efforts of West Lake Landfill Areas 1 and 2 in a letter dated July 6, 2015. The Work Plan was based on, and incorporated procedures from other work plans previously submitted to, and approved by, the EPA. The previously submitted CSWP included various requirements of a quality assurance project plan (QAPP) for sample collection and plan-specific measurements and analyses for work to be conducted in the field at West Lake Landfill. The scope of work that is discussed in the Work Plan that is covered by this QAPP addendum comprises the additional laboratory analytical testing to obtain site-specific data for use in fate-and-transport evaluations that were previously requested by the EPA.

As discussed in the Work Plan, additional testing is designed to identify and distinguish the chemical composition of the materials containing radionuclides and the speciation of the radionuclides in these materials, and to provide data to parameterize a geochemical fate-and-transport model. Specifically, two samples will be collected from each of four borings in Area 1, and two samples from each of six borings in Area 2 (resulting in a total of 20 solid samples). Consistent with prior sample collection activities at the West Lake Landfill that targeted RIM, the first sample obtained from each boring will be selected from a depth interval that displays high gamma readings (based upon gamma scans of the borehole and/or core samples). Analytical data from these samples will be used to evaluate the geochemistry, stability and leachability of any radionuclide occurrences in Areas 1 and 2. The second sample will be collected from a deeper interval that does not display elevated gamma readings. Analytical data from these samples will be used to evaluate potential attenuation of radionuclides that may be mobilized from the overlying RIM. Samples will be placed in polyethylene bags, vacuum-sealed, frozen and subsequently shipped to the laboratory on dry ice in order to preserve the in-situ chemical oxidation state of the samples. Also, prior to analysis, samples will be air-dried and homogenized by the laboratory in an anoxic glove box.

Table 1 presents both a summary of the proposed laboratory analyses to be performed in support of the fate-and-transport evaluations and the intended use of the data from each of the tests.

Table 1. Proposed Solids Testing to Support Fate and Transport Evaluations

Fate and Transport Model Input Parameter	Description	Number of Samples			
		Radiological Soils	Non-Radiological Soils	Replicate	TOTAL
Radionuclide Concentrations	Ra-226; Ra-228; Th-230; Th-232; U-234, U-235; U-238	10	10	1	**21**
Major Cations and Anions	Barium, Calcium, Iron, Magnesium, Manganese, Potassium, Sodium, Sulfate, Carbonate, Fluoride, Phosphate	10	10	1	**21**
pH & Redox Indicators	Sulfide, Iron(II), Iron(III), Uranium(VI), pH	10	10	1	**21**
Organic Carbon Content	Total Organic Carbon (TOC)	10	10	1	**21**
Major Minerals	X-Ray Diffraction (XRD)	10	10	1	**21**
Radionuclide Speciation	Sequential Extraction Analysis [1]	10	--	1	**11**
Mineral Reactivity	Scanning Electron Microscope with Energy Dispersive X-ray Spectrometry (SEM/EDS)	4	--	--	**4**
Attenuation Capacity	Cation Exchange Capacity (CEC)	10	10	1	**21**
Leachate Composition	Sequential Batch Leaching Test (SBLT)[2]	10	--	1	**11**
	Sythetic Precipitation Leaching Procedure (SPLP) Test (EPA Method 1312; pH 5.0)	--	10	1	**11**

[1] See Table 2 for a description of the sequential extraction tests.
[2] SBLT consists of 6 extractions using methodology of SPLP Test (L:S of 20:1). Extractions 1-3 use 0.05 M NaCl + 1000 mg/L humic acid (HA) at pH 7.0. Extractions 4-6 based on SPLP Test at pH 5.0 (EPA Method 1312). All extractions analyzed for U, Th, Ra, pH, cations, anions, and DOC.
[3] SPLP (pH 5; EPA Method 1312) to analyze for major cations and anions, pH, and DOC

In summary, samples to be tested for fate-and-transport related parameters will be subject to the following analyses, for reasons highlighted in the following bullets:

- Uranium, thorium, and radium isotopes;
- Major cations and anions (including calcium, magnesium, manganese, sodium, potassium, barium, carbonate, sulfate, fluoride and phosphate);
- pH and redox indicators (Fe(II), Fe(III), sulfide, and U(VI));
- Total organic carbon (TOC), which assesses the levels of humic and fulvic acids that affect partitioning and mobility of radionuclides (and the longevity of potentially-reducing conditions within the landfill);
- X-Ray Diffraction (XRD), which quantifies the abundance of major minerals (e.g. barite and/or calcite in the waste) that potentially-affect leachate composition and radionuclide speciation (XRD provides a semi-quantitative description of the primary

minerals present in a sample to corroborate the calculated mineralogy based on cation and anion analyses);

- Sequential extraction analysis, which consists of sample digestion in a series of sequential extraction steps designed to dissolve specific minerals, will be used to access the speciation of radionuclides in the samples. Please see Table 2 for details of the analysis.
- Scanning Electron Microscope with Energy Dispersive X-ray Spectrometry (SEM/EDS), which provides a semi-quantitative method for elemental mapping (e.g., barite, gypsum, calcite, and oxides);
- Cation-Exchange-Capacity (CEC), which estimates the potential capacity of the waste/soil to adsorb radionuclides; and,
- Synthetic Precipitation Leaching Procedure (SPLP) tests and sequential SPLP tests (called sequential batch leaching tests or SBLT in Table 1), which will primarily be used to evaluate the parameterization of the fate and transport model by comparing measured and simulated SBLT leaching test results.

Table 2. Sequential Extraction Procedure for Characterizing Source Materials

Step	Targeted Phases	Reagant
1	Soluble / Exchangeable: Exchangeable ions	10 mL of 1 M $Mg(NO_3)_2$, pH 7, 4 hr, 25 °C + 1 water wash (10 mL)
2	Acid Soluble: Carbonates	25 mL of 1 M CH_3CO_2Na, pH 5, 6 hr, 25 °C + 1 water wash (10 mL)
3	Organics/Sulfides: Humic materials and Fe-sulfides	30 mL of 0.1 M $Na_4P_2O_7$, pH 10, 20 hr, 25 °C + 1 water wash (10 mL)
4	Amorphous Oxides: Mn-oxides, ferrihydrite, and secondary U minerals	10 mL of 0.2 M $(NH_4)_2C_2O_4$, pH 3, 4 hr, 25 °C (dark) + 1 water wash (10 mL)
5	Crystalline Oxides: Goethite and Magnetite	25 mL of 0.2 M $(NH_4)_2C_2O_4$ in 0.1 M ascorbic acid, pH 3, 0.5 hr, 95 °C + 1 water wash (10 mL)
6	Alkaline-earth sulfates: Barite	200 mL of 0.11 M Na_2EDTA + 1.7 M NH_4O_4, 4 hr, 95 °C + 1 water wash (10 mL)
7	Residual: Clays, primary U- and Th-oxides	HF-$HClO_4$ (Complete digestion)

Notes: Method based on Liu et al. (2011). All extractions use 1 gram of solid and all solutions analyzed for U, Ra, Th, pH, Fe, Mn, Ca, Ba, total carbon and sulfur; Procedure includes digestion/centrifugation, wash/centrifugation and analysis steps. Finally, steps 1 and 2 will be conducted in an anoxic glovebox.

Section 3
Quality Objectives and Criteria

Data quality objectives (DQOs) are qualitative and quantitative statements that define the type, quality, and quantity of environmental data appropriate for the intended application. The overall DQO for this project is the obtain data of sufficient quality to allow for the evaluation of the fate and transport of site contaminates of concerns, primarily RIM. Specific objectives of the proposed analyses are described in the following paragraphs.

The objective of solid-phase testing is to identify radionuclide and inorganic constituents that occur above background levels. Therefore, typical background concentrations in soils and landfill materials are used as the target levels. For radionuclides, the target concentrations are based on the background concentrations listed in Tables 6-1 through 6-4 of the RI Report (EMSI, 2000). Target concentrations for solid and aqueous extracts are reported in Table 3. For the other constituents listed in Table 3, the target concentrations are based on average background concentrations found in United States soils (Dragun and Chiasson 1991).

The objective of sequential extraction tests is to identify the speciation of select constituents between operationally-defined phases. Therefore, the reported target concentrations in Table 4 (below) are based on the goal of detecting a 1% recovery of any particular analyte in each sequential digestion. Since only radiological materials will be submitted for sequential extractions, the reported values in Table 4 for radionuclides are based upon a 1% recovery from surface and subsurface landfill samples (Tables 6-1 through 6-4 of the RI Report). Target concentrations for inorganic constituents are based on a 1% recovery of background soils (using the soil concentrations reported in Table 3).

The Synthetic Precipitation Leaching Procedure (SPLP) tests and sequential SPLP tests (called SBLTs in this QAPP) are designed to provide data that can be used to calibrate the fate-and-transport model to data and conditions obtained from and encountered in the field site. However, because the sequential SPLP tests additionally provide an estimate of leachate composition from radiological materials, the target concentrations used for radionuclides in Table 4 are the EPA maximum contaminant levels. For the other constituents, the same target concentrations as the sequential extraction tests are proposed.

The primary procedural tools required to achieve these DQOs are:

- Application of analytical methods with detection limits suitable to meet this DQO;
- Sampling methodologies to prevent cross-contamination and sample degradation;
- Collection of suitable laboratory and field duplicates and blanks; and
- Decontamination methods to prevent cross-contamination of samples.

Analytical performance requirements for this work are expressed in terms of precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS).

Table 3. Target Analytes, Methods, Reporting Limits and Target Concentrations for Soil Samples

Analysis	Parameter	Method Reference	Laboratory Soil Samples		Target Concentration[†]
			Units	Reporting Limit	
Radionuclides	Ra-226	EPA 900.0 Mod (AP-006)	pCi/g	1	1.3
	Ra-228	EPA 904.0 Mod (AP-007)	pCi/g	1	2.37
	Th-230	HASL EML Th-01 Modified (AP-005)	pCi/g	1	2.45
	Th-232	HASL EML Th-01 Modified (AP-005)	pCi/g	1	1.55
	U-234	EPA 6020	mg/Kg	0.001	2.73
	U-235	EPA 6020	mg/Kg	0.01	1.15
	U-238	EPA 6020	mg/Kg	1	2.24
Major Cations and Anions	Barium	EPA 3050, EPA 6010	mg/Kg	2	580
	Calcium	EPA 3050, EPA 6010	mg/Kg	20	24000
	Carbonate	Water Leach, SM 2320E	mg/Kg	per method	25000
	Fluoride	Water Leach, EPA 300	mg/Kg	2	430
	Iron	EPA 3050, EPA 6010	mg/Kg	20	26000
	Magnesium	EPA 3050, EPA 6010	mg/Kg	20	9000
	Manganese	EPA 3050, EPA 6010	mg/Kg	20	550
	Phosphate	Water Leach, EPA 300	mg/Kg	12	430
	Potassium	EPA 3050, EPA 6010	mg/Kg	20	15000
	Sodium	EPA 3050, EPA 6010	mg/Kg	20	12000
	Sulfate	Water Leach, EPA 300	mg/Kg	12	4800
Redox Indicators	Ferric Iron	EPA 6010 by difference with Ferrous	mg/Kg	TBD	26000
	Ferrous Iron	SM 3500-Fe B	mg/Kg	TBD	26000
	Sulfide	EPA 6010 / EPA-OW-OST 376.3 **	mg/Kg	TBD	1700
	U(VI)	MCL-7737	mg/Kg	TBD	2.7
General Chemistry	Cation Exchange Capacity	EPA 9081	meq/100g	per method	--
	pH	EPA 9045D	s.u.	0.05	0.1
	Total Organic Carbon	EPA 9060/9060A	wt. %	0.01	>1
Non-Destructive Testing	X-Ray Diffraction	MCL-7712	wt. %	3-5	5
	SEM/EDS****	MCL-7708	wt. %	0.1	0.1

"–" = Not Requested or Not Applicable

TBD = To be determined, dependent on the spike recovery in the sample

per method = the reporting limit is dependent on the nature of the sample (carbonate on the pH; CEC depends on cation concentrations)

** Sulfide determination will be difference between total sulfur by EPA 3050 / 6010 and preparation for acid volatile sulfur EPA 376.3 / EPA 6010

**** SEM will be used instead of EMPA; SEM/EDS provides mineral identification and a semi-quantitative method for elemental mapping, as needed.

[†] Target concentration for radionuclides based on minimum background reported in RI Report; Other inorganics are average background reported for U.S. soil (Dragun and Chiasson 1991)

Eberline AP-005 is for Isotopic Thoirum, AP-005 is for Radium-226 and AP-007 is for Radium-228. Soil preparation procedures are Eberline AP-001 & AP-002

Table 4. Target Analytes, Methods, Reporting Limits and Target Concentrations for Aqueous Extractions

Analysis	Parameter	Method Reference	Sequential Extractions			SPLP Extractions		
			Units	Reporting Limit	Target[†,+]	Units	Reporting Limit	Target[+,x]
Radionuclides	Ra-226	AP-006	pCi/L *	10	100	pCi/L	5	5
	Ra-228	AP-007	pCi/L *	10	100	pCi/L	5	5
	Total Radium	***	pCi/L *	10	100	pCi/L	5	5
	Total Thorium	HASL EML Th-01 Modified	pCi/L *	10	10000	pCi/L	5	5
	Total Uranium	EPA 6020	mg/L *	1	300	mg/L	0.03	0.03
Major Cations and Anions	Barium	EPA 6010	mg/L *	0.2	0.6	mg/L *	0.2	0.6
	Calcium	EPA 6010	mg/L *	2	24	mg/L *	2	24
	Carbonate	SM 2320E	mg/L	per method	25	mg/L	per method	25
	Iron	EPA 6010	mg/L *	2	26	mg/L *	2	26
	Magnesium	EPA 6010	mg/L	2	9	mg/L	2	9
	Manganese	EPA 6010	mg/L *	0.2	0.6	mg/L *	0.2	0.6
	Potassium	EPA 6010	mg/L	2	15	mg/L	2	15
	Sodium	EPA 6010	mg/L	2	12	mg/L	2	12
	Sulfate	EPA 300	mg/L *	6	6	mg/L *	6	6
General Chemistry	pH	EPA 150.1	s.u.	0.05	0.1	s.u.	0.05	0.1
	DOC	EPA 9060/9060A	mg/L *	--	--	mg/L	1	1

"--" = Not Requested or Not Applicable

per method = the reporting limit is dependent on the nature of the sample (carbonate on the pH)

* Calculated based on sequential extraction procedure Step 1, which is the minimum volume used (reporting limits will be lower for other extraction steps and SPLP-based tests)

*** Total Radium will be determined by summing the results from Radium-226 and Radium-228.

[†] Target concentrations for radionuclides based on 1% recovery in individual extractions from radiological samples (assumed Ra = 100 pCi/g; Th = 10,000 pCi/g; U = 100 pCi/g; RI Report)

[+] Target concentration for inorganics in sequential extractions and SPLP tests based on 1% recovery in individual extractions of background soils

[x] Target radionuclide concentrations in SPLP tests based on EPA MCLs

3.1 Precision

Precision is a measurement of the degree of agreement of replicate data, which is quantitatively assessed based on the relative percent difference. The relative percent difference (RPD) is calculated with the following equation:

$$RPD = \frac{A - B}{\frac{A + B}{2}} \times 100$$

where:
A = The larger of the two values.
B = The smaller of the two values.

For this project, generalized acceptance criteria of 35% RPD will be used for the samples. Failure to achieve a value below these levels will require a review of the field and laboratory documentation for these samples to assess possible causes, and may result in corrective actions.

3.1.2 Field Precision

Field precision will be assessed through the collection and measurement of field duplicate samples. Duplicate samples will be analyzed for a minimum of 5% of samples to check for overall variability introduced by sample heterogeneity, sampling and analytical procedures.

3.1.3 Laboratory Precision

Laboratory precision is assessed through the calculation of relative percent differences (RPDs) for two replicate samples. The precision of the analysis can be inferred through the use of one of the following: laboratory control duplicate samples; matrix spike and matrix spike duplicate (MS/MSD) samples, or unspiked duplicate samples. The laboratory analyzes one or more of these duplicate samples at a standard rate per type of analysis, depending upon the analytical method.

3.2 Accuracy

Accuracy is the degree of agreement between a measurement or observation and an accepted value.

3.2.1 Field Accuracy

Field accuracy is assessed through the adherence to all sampling handling, preservation, and holding time requirements. Accuracy of field instruments will be assessed by instrument calibration and calibration checks. These calibration checks will be documented in field notes.

3.2.2 Laboratory Accuracy

Laboratory accuracy is assessed by the analysis of matrix spikes (MS) and laboratory control samples (LCS). The results are expressed as a percent recovery. Surrogate recoveries may also be used to assess accuracy. Method blanks are used to assess contamination resulting from

laboratory procedures. Laboratory control samples, method blanks, and preparation blanks will be analyzed at a standard frequency per type of analysis.

The percent recovery (% R) is calculated with the following equation:

$$\% R = \frac{A - B}{C} x\ 100$$

where:
A = The analyte concentration determined experimentally from the spiked sample.
B = The background level determined by a separate analysis of the unspiked sample.
C = The amount of the spike added.

3.3 Representativeness

Representativeness is a qualitative measure of the degree to which sample data accurately and precisely represent a characteristic environmental condition. Representativeness is a subjective parameter and is used to evaluate the efficacy of the sampling plan design. Representativeness is demonstrated by providing full descriptions of the sampling techniques and the rationale used for selecting sampling locations in the project planning documents. The measure of representativeness is answered during the preparation of the sampling and analysis approach and rationale, and then reassessed during the data usability process.

The sampling and analysis approach and rationale are defined in the Work Plan and in the CSWP.

3.4 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was planned to be obtained under normal conditions. Percent completeness is calculated with the following equation:

$$\% \ Completene\ ss\ = \frac{Valid\ \ Data\ \ Obtained}{Total\ \ Data\ \ Planned} \text{ x } 100$$

Experience on similar projects has shown a reasonable goal considering laboratory performance is 80 percent completeness. If sufficient valid data are not obtained, corrective action will be initiated by the Project Manager to modify field, data shipping, or laboratory procedures.

3.5 Comparability

Comparability expresses the confidence with which one data set can be compared with another data set obtained during parallel or previous investigations. Comparability can be related to precision and accuracy as these parameters are measures of data reliability.

Chemical samples from the same media are generally considered comparable if the same

procedures for collecting and analyzing the samples are used, if the samples comply with the same Quality Assurance/Quality Control (QA/QC) procedures, and if the units of measurement are the same. Comparability in this project is addressed through the use of uniform analytical methods, sampling procedures and field procedures across the entire site.

3.6 Sensitivity

Sensitivity is the measure of the concentration at which an analytical method can positively identify and report analytical results. The sensitivity of a given method is commonly referred to as the detection limit. Although there is no single definition of this term, the following terms and definition of detection limits will be used:

- Instrument detection limit (IDL) is the minimum concentration that can be measured from instrument background noise under ideal conditions.
- Method detection limit (MDL) is a statistically determined concentration. It is the minimum concentration of an analyte that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero as determined in the same or a similar matrix. Because of the lack of analytical precision at this range, sample results (if reported by the laboratory) greater than the MDL but less than the reporting limit (RL) would be qualified as "estimated".
- Reporting limit (RL) is the concentration of the target analyte that the laboratory has demonstrated the ability to measure within specified limits of precision and accuracy during routine laboratory operating conditions. This value is variable and highly matrix dependent. It is the minimum concentration that will be reported as unqualified by the laboratory.

For sensitivity, the quality objective is to analyze data with a method that achieves RLs that are below or equal to the task-specific target analysis goals or concentrations. Planned sensitives are presented in Tables 3 and 4.

Section 4
Special Training/Certification

As discussed in the approved CSWP quality assurance section, there are no special training/certifications required for this scope of work.

Section 5
Documentation and Records

Documentation and records are detailed in Section 9 of the approved CSWP.

Section 6
Sampling Process and Design

Specific methods and designs for sample collection and analysis, handling, reporting and other aspects of sample collection are detailed in Section 4 of the approved CSWP.

Section 7
Sample Handling and Custody

Specific methods and designs for sample handling and custody are detailed in Section 4 of the approved CSWP.

Section 8
Analytical Methods

This section provides information on the analytical methods for each medium of concern. In general, all analyses will utilize EPA-approved methods or other recognized standard methods. Method references for laboratory analyses that may be performed for the anticipated work are provided in Table 1. Sample size, preservation requirements and holding times for analytical parameters not detailed in the CSWP are summarized in Table 5.

The primary laboratory for this study will be Materials & Chemistry Laboratory, Inc. (MCL) of Oakridge, TN. The primary laboratory's Quality Assurance Manual, and internal analytical Standard Operating Procedures are included as Attachment 1.

Laboratory turnaround times for this project shall generally range between 8 and 12 weeks.

Table 5 Sample Requirements and Holding Times

Sample Type	Weight Requested	Preservation	Hold Time for Radiological Analyses	Container
Radioactive Soil	250 grams	<6°C	180 days	Polyethylene Bag *
Non Radioactive Soil	1000 grams	<6°C	180 days	Polyethylene Bag

*Secondary containment required for radiological samples

Section 9
Quality Control

Quality control is discussed in Section 4.9 of the approved CSWP.

Section 10
Data Management

Data Management is discussed in Section 9 of the approved CSWP.

Section 11
Assessment and Response Actions

This section presents the internal and external checks (assessments) that have been built into this project to assure that:

- Elements of this QAPP addendum have been correctly implemented as prescribed for all tasks this project;
- The quality of the data generated is adequate and satisfies the DQOs that have been identified in this QAPP addendum; and
- Corrective actions, when needed, are implemented in a timely manner and their effectiveness is confirmed.

Assessment activities may include surveillance, inspection, peer review, management systems review, readiness review, technical systems audit, performance evaluation, and data quality assessment. Assessment and response actions are detailed in the approved CSWP.

Section 12
Data Review, Verification, Validation, Usability & Reconciliation

This section provides a description of the QA activities that will occur after the data collection phase of the project is completed. Implementation of this section will determine whether or not the data conform to the specified criteria, thus satisfying the project objectives.

Data Review, Verification, Validation, Usability & Reconciliation is discussed in Section 8.3 of the approved CSWP.

Citations

Dragun, J., and A. Chiasson. 1991, Elements in North America. Hazardous Materials Control Resources Institute, Greenbelt, MD.

Engineering Management Support, Inc. (EMSI), 2000. Remedial Investigation Report, West Lake Landfill Operable Unit 1. Prepared for West Lake OU-1 Respondents Group. April 10, 2000.

Engineering Management Support, Inc. (EMSI), 2015. Work Plan for Additional Characterization of Extent of Radiologically-Impacted Material in Areas 1 and 2, West Lake Landfill Operable Unit-1, Bridgeton, Missouri.

Feezor Engineering., Inc. (FE), 2014. Core Sampling (Phase 1B, 1C, and 2) Work Plan – Revision 1 (CSWP). Prepared for Bridgeton Landfill, LLC. January, 2008.

Liu, D.J. and M.J. Hendry, 2011. Controls on 226Ra during Raffinate Neutralization at the Key Lake Uranium Mill, Saskatchewan, Canada. Appl. Geochem. 26: 2113-2120.

ATTACHMENT 1

MATERIALS CHEMISTRY LABORATORY, INC. MCL QUALITY ASSURANCE PLAN

MCL INC
MATERIALS & CHEMISTRY LABORATORY, INC.

"Linking Technology to Solutions"

Quality Assurance Plan
MCL-7701

Materials and Chemistry Laboratory, Inc.
East Tennessee Technology Park
Building K-1006
2010 Highway 58, Suite 1000
Oak Ridge, Tennessee 37830

Issued
Revision 13
Revision 13.1, December 6, 2013
Revision 13.2, April 7, 2014
Revision 13.3, November 24, 2014
Revision 13.4, February 5, 2015
Revision 14, September 2015

Controlled Copy No.

Table of Contents

Appendices

1.0 ORGANIZATION

1.1 Introduction

Materials and Chemistry Laboratory, Inc. (MCLinc) provides technical support to a variety of customers and programs. Work done may be classified at levels up to U.S. Department of Energy (DOE) "Q" or "QX" (S-RD [level: secret – Category: Restricted Data]), and may involve radioactive, special nuclear materials (SNM), and/or hazardous materials. Scope of work includes, but is not limited to, characterization studies, research projects, development efforts, lab-to-bench-to-pilot scale processes, process optimization, and methodology development. Quality is inherent in all aspects of MCLinc work. This plan and the references herein, ensure that a management framework is defined for the establishment of quality MCLinc practices.

It is noted that this plan does not specifically address all aspects of the Industrial Hygiene Analysis Laboratory (IHL) within MCLinc. The IHL is an American Industrial Hygiene Association Program (AIHA) Laboratory Accreditation Program (LAP), LLC (AIHA) accredited laboratory. The latest AIHA assessment was done under the requirements of International Organization for Standardization/International Engineering Consortium (ISO/IEC) 17025 international standard. The IHL operates under a stand-alone quality plan, MCLinc, "Industrial Hygiene Laboratory Quality Assurance Manual," MCL-7719. The information contained within the MCLinc Quality Assurance Plan (QAP) will still apply to the overall operation of all IHL functions, but will not specifically address some of the AIHA-required details that are unique to the IHL. This document and other supporting Standard Operating Procedures (SOPs) will apply to all MCLinc AIHA accreditations.

1.2 Quality Assurance Policy

The MCLinc Quality Assurance (QA) Policy approved by the MCLinc President is to assure that the *QA* practices utilized by the MCLinc staff conform to requirements, standards, and responsibilities necessary for maintaining a quality organization in conjunction with DOE, and customer-based expectations. This policy incorporated into this QAP will help to minimize the risk and environmental impact of processes influenced and performed by MCLinc as well as maximizing the safety, reliability and performance of MCLinc methodologies and practices. The MCLinc QA Policy must be

rigid to assure quality objectives are met, but also dynamic by having procedures in place to allow continual improvement in the quality management process (annual assessment, SOP change procedure, and corrective action procedures are examples of means to allow improvement).

This QAP is designed to specifically meet the management and technical requirements for testing facilities of the internationally recognized standard ISO/IEC 17025 and American Society of Mechanical Engineer, Nuclear Quality Assurance, Level 1 (ASME NQA-1), National Environmental Laboratory Accreditation Conference related U.S. Environmental Protection Agency (EPA) and DOE documents noted in the Reference Section. See Appendix A for a cross-reference table, by section of this QAP, to the specific requirements of ISO/IEC 17025, Title 10, Code of Federal Regulations (10 CFR) Part 830.120, American National Standards Institute/American Society for Quality Control (ANSI/ASQC) E4-1994, and ASME NQA-1. Changes to this document may only be made with the approval of the MCLinc President and Quality Assurance Officer (QAO) per the SOP "Document Control", MCL-7703.

1.3 Organizational Responsibilities

The organizational structure of MCLinc is shown in Appendix B. MCLinc must be able to maintain flexible work responsibilities to ensure that a wide variety of customer, Site (East Tennessee Technology Park and URS/CH2M Oak Ridge, LLC and Landlord (DOE and Community Reuse Organization of East Tennessee [CROET]) requirements can be met.

1.4 Functional Responsibilities

The MCLinc President provides daily guidance and administrative support to the MCLinc staff and is committed to ensuring compliance to this QAP and ISO/IEC Standard 17025, AIHA, and other quality requirements of our customers. The MCLinc President is supported by the Technical Director (TD) and the Laboratory Manager (LM). These positions provide routine assistance to personnel and customers on the capabilities and application of MCLinc resources to solving problems.

The TD has responsibility to provide technical direction to the Project Manager (PM) and technical assistance to our clients. The LM has responsibilities for the day-to-day operation of the laboratories and to make sure resources are available to meet the needs of our clients and this plan. The Quality Assurance Specialist (QAS) performs quality duties as assigned by the QAO. The QAS is the designated alternate for the QAO in his absence.

All staff members of MCLinc are responsible for ensuring that customer objectives (i.e., quality, time frame, budget, applicability) are met in accordance with this plan and any other applicable documentation. The work performed by any and all staff members is necessary to meet our management quality objectives and those of our clients. A staff

member is empowered to stop work due to any safety issue or when the quality of the product is endangered and report such concerns to the QAO. Appendix C lists MCLinc personnel and their support functions which will help ensure the quality objectives of MCLinc and its customers are met. The QAO is the point of contact for the implementation and enforcement of quality-based procedures. Conflicts in operating methods and procedures will be resolved by the QAO with support from the appropriate management personnel. The QAO is committed to compliance to this QAP and ISO/IEC Standard 17025 and other quality requirements of our customers. The QAO, as necessary, develops and issues SOPs or QA Directives in memo format to further define or explain items covered by this QAP or other areas needing procedures defined.

The QAO has the authority to stop work at any time to assess a reported problem or investigate a quality system failure or trend.

The PM is either self-appointed or is selected by the LM or MCLinc President based upon the nature of the project. The PM is the person responsible for the control and coordination of all activities associated with the successful completion of a customer task.

1.5 Facility/Security Attributes

MCLinc is leasing the K-1006 facility from the DOE, through CROET as part of the DOE reindustrialization initiative. The brick facility is approximately 28,000 square feet (ft^2). The area available to MCLinc under its lease with the CROET is approximately 25,300 ft^2. The facility is designed to be a laboratory facility. The second floor contains office space (15 rooms) and one storage closet. The first floor accommodates approximately: 30 labs, 12 offices, 5 administrative/common areas, 2 maintenance areas, 5 hallways, and 2 utility chases. There is approximately 12,445 ft^2 of laboratory space and 12,885 ft^2 of non-laboratory space within the MCLinc facility. The facility is dedicated to handling virtually any type of environmental contaminant and is operated by a multidisciplinary staff qualified to address technical issues pertaining to radiological and hazardous materials.

Although MCLinc is a private commercial entity, it still has some operational restrictions based upon where and what type of business it does. As part of the basis for MCLinc to continue to be authorized to perform classified work, a graded approach to physical security had to be implemented. The physical security, and hence the main basis for the security infrastructure of the MCLinc facility, is based upon three to four layers of access control. The first layer is that access to the site is monitored by Site Security with all visitors required to go through the security office. The second layer is the controlled access requirements (Hirsch badge reader system or controlled access key) to gain access into building K-1006. The third layer is controlled key access into laboratory areas. The fourth layer is the controlled access storage area within various laboratories. The Facility Security Officer (FSO) will assign keys. All assigned keys will either be physically controlled by the individual person or will be controlled by that individual using a unique

lock/combination controlled storage area. Lost keys will be reported to the FSO immediately. This graded approach to physical security provides the required control over facility access for security issues as well as for proper chain of custody (COC) of certain materials and samples.

1.6 Commitment to Quality

The managers, owners and employees of MCLinc, an employee-owned company, are committed to a policy whereby all personnel are free of any undue internal or external commercial, financial, or other pressures and influences which may adversely affect the quality of the work. Any staff member feeling such pressures shall report this concern immediately to the LM, TD, QAO or the MCLinc President for investigation and corrective action.

As noted under the Facilities Section, the facility is secure and the staff is knowledgeable in the handling of confidential information for the DOE. This same approach is extended to all clients in that client confidential information or proprietary rights are maintained in confidence and all such documents or electronic files are stored in locked files or in computers password-protected and accessible only to authorized staff. See Section 5.5, Classified Materials. The MCLinc reputation and success depends on the high integrity of the staff. MCLinc's policy is that technical and business competence, impartiality, judgment, and data quality and operational integrity must be maintained at all times. These elements are key to maintaining the quality of our efforts. The employees therefore must be aware of their contributions to maintaining the management quality system.

The MCLinc management staff has the responsibility and authority to provide the resources to complete the above and through staff and project meetings and other communication tools (i.e., e-mail) encourage the staff to communicate their assessment of the management system. The MCLinc management also has the responsibility for training, implementation, maintenance and improvement of the management system and to identify and correct variances from the system. The QAO, TD, LM, PM and QA assessments are key in identifying any variance from this policy which must be investigated and corrective action taken including disciplinary action.

2.0 MANAGEMENT SYSTEMS

2.1 Standards and Reference Materials

MCLinc has a need for a variety of standards and reference materials. Where possible, these standards and reference materials must be purchased from an ISO/IEC 17025; 2005 certified vendor. Traceability of these standards must also be demonstrated on the Certificate of Analyses of the standard. The standards and reference material must be handled, stored and used per the manufacturer's specification to avoid contamination and deterioration.

Many standard methods require use of second source standards to check primary standards (i.e., organics and metals). The "Quality Systems for Analytical Services" (QSAS) requires radiation calibration standards to be verified prior to use and annually as follows:

- At least three verification measurements of a standard are used to determine the mean value and standard deviation of verification results.
- Mean value is within 5 percent (%) of certified value.
- Two sigma deviations is less than 10% of the mean value.

If specifications are not met, corrective actions must be evaluated and implemented.

2.2 Calibration

Instrument and equipment performance evaluation, maintenance, and documentation are the responsibility of the instrument owner. Appendix D lists the responsible owner and authorized operator for the major instrumentation with the MCLinc facility. These instruments have specific QA documents that outline the minimum calibration requirements. For the general or common data acquisition laboratory equipment (e.g., balances, pH meters,) the "Calibration, Maintenance and Inspection Plan," MCL-7711, outlines the calibration and documentation requirements for those components which may influence the work being performed. When there is a need for outside calibration of laboratory equipment, the vendor/material used must be traceable to national standard setting bodies such as National Institute of Standards and Technology (NIST) or ISO approved.

2.3 Facility Maintenance

The housekeeping and maintenance of each MCLinc office or laboratory facility is the direct responsibility of all MCLinc personnel. MCLinc facilities should be kept clean and orderly and the temperature and humidity controlled to meet the needs of the testing instrument. If the MCLinc staff member responsible for an area is temporarily or permanently unable to comply with this standard, he or she should advise management of the problem. Specific health and safety requirements are outlined in the "Chemical Hygiene Plan," MCL-7702 and the "Health and Safety Plan," MCL-7717. Specific requirements for maintenance and facility documentation are provided in the "Calibration, Maintenance and Inspection Plan," MCL-7711.

2.4 Work Environment

MCLinc maintains a safe and clean working environment. All laboratory areas and materials are maintained in a clean and orderly fashion to ensure the work performed will not be compromised by the local environment of the laboratory facility. The MCLinc personnel performing work are responsible for ensuring that all cleanliness requirements are met prior to commencing work. The "Chemical Hygiene Plan," MCL-7702, provides additional detail.

2.5 Laboratory Supplies

These materials are stored and controlled based upon the hazardous nature of the material. Individual personnel are responsible for ensuring that the integrity of the laboratory supplies is adequate to meet MCLinc and the customer's expectations. The ordering, reporting and tracking of chemicals is addressed in the "Chemical Hygiene Plan," MCL-7702 and the "Procurement Control Plan," MCL-7727. The ordering, reporting, and tracking of radiological materials are addressed in the "Implementation SOP for the Radiation Protection Plan," MCL-7715.

2.6 Special Nuclear Materials (SNM)

The control and monitoring of SNM is detailed in "Nuclear Materials Control and Accountability Plan," MCL-7706. The Radiological Safety Officer (RSO) is responsible for oversight and control of SNM.

2.7 Material and Sample Receipt

Samples and materials are received at the MCLinc facility from various sources and require various levels of oversight and control. Sample login, tracking, documentation, archival, disposal, and/or return procedures are detailed in "Project Management Plan," MCL-7704 and Operator Aid Appendix O in MCL-7756, "Operator Aids." This procedure provides guidance on issues such as non-conformance reports (NCR), cross contamination, inspection log sheets, sample tracking and management, and laboratory records associated with sample management. The "Procurement Control Plan," MCL-

7727 and Section 3.1 provide details on the quality control (QC) checks and documentation required for receipt of materials.

2.8 Controlled Samples

Controlled samples have COC documentation. COC forms can either be supplied by the customer or by MCLinc. The COC ensures that the samples will be maintained within the MCLinc facility. The COC form will be documented to reflect when the samples either leave the MCLinc facility or a non-MCLinc employee is provided direct, unsupervised, access to the samples. No internal COC control is required for samples remaining within the MCLinc facility.

2.9 Non-COC Samples

Many projects that are performed by MCLinc are on samples that do not have an associated COC and are typically representative of a process or condition that does not require COC control. These samples are maintained within the secure MCLinc facility. The use and control of these samples is the responsibility of the PM. The PM may elect to document any special handling or storage protocols that should be used for a given sample or group of samples. The PM is responsible for ensuring that proper documentation and labeling is provided to ensure that any MCLinc employee that may need to utilize these materials understands the specific requirements associated with the samples.

2.10 Sampling and Sample Preparation

In MCLinc projects where actual sampling of the process is required, the details of the sampling process and procedures to follow must be defined in a project sampling plan or scope of work. All samplers must be trained on the procedures and understand the critical importance of the sample to the project. The resultant sample must represent the source being sampled and the PM must define steps to be taken to best approximate a homogenized sample. This is also a critical step in sub-sampling samples received at the laboratory for testing.

The staff member responsible for the analysis shall determine sample preparation techniques utilized. Sample preparation techniques shall be documented. Good laboratory notebook protocols will be used when documenting the laboratory, data, and/or project activities. Additional quality, safety, and environmental aspects of sample preparation are provided in, "Sample Preparation Plan," MCL-7710.

2.11 Instrumentation and Maintenance

MCLinc has a variety of laboratory instrumentation ranging from very complex (e.g., transmission electron microscope) to standard laboratory instrumentation (e.g., pH meter). The level of the documentation required for the standardized use of instrumentation is decided by the LM. The instrumentation listed in Appendix D requires

some level of QA and/or operational guidance. The use and control of the general laboratory equipment is provided in "Calibration, Maintenance and Inspection Plan," MCL-7711, technology specific SOPs (e.g., MCL-7708, "Electron Microscope Operations Plan"), project specific QA plans, vendor manuals, and other customer specific documentation. MCLinc staff members using an instrument are responsible for documenting non-routine maintenance and repairs, and maintaining an inventory of consumables and commonly needed parts.

2.12 Quality Control Samples and Assessment of Data

Since the vast majority of the projects performed by MCLinc are non-routine or the application of a routine procedure to a non-routine use, the measurement quality objectives vary significantly. The basic objective of all MCLinc measurements/analyses are to assure: (1) the procedure measures the parameter of interest, (2) the instrument/system is calibrated and operating properly, (3) the sample was properly prepared and handled in a way to minimize contamination, and (4) the data is calculated, reviewed and reported properly. Depending on the procedure or technique utilized MCLinc achieves the above by using QC samples. These QC samples include one or more of the following:

- Method Blank
- Instrument Blank
- Calibration Check Standard
- Laboratory Control Sample's (Duplicates)
- Matrix Spike and/or Matrix Spike Duplicate
- Duplicate Sample
- Certified Sample

In many cases the QC sample requirements, if not defined by the procedure, are defined by the client and MCLinc at time of project inception. Any anomalies or failures of QC samples must be evaluated and if persistent reported as a non-conformance requiring corrective action.

The laboratory control samples (LCSs) shall be used by the analyst to evaluate method performance. In cases where the method is run infrequently (less than 20 samples per month), the analyst shall evaluate LCS recoveries against criteria in the method or use a default of 100% ± 25% recovery. Corrective action will include rerun of the LCS and if it still fails evaluation by the QA*O* verses client project needs.

For analytical methods requiring LCSs and run frequently (more than 20 samples per month), LCS data shall be tabulated for review by the analyst to see trends with calculation of the standard deviation (sigma). The data points generated for each sample set should be evaluated as follows:

± 1 Sigma shall contain 2/3 of the points
± 2 Sigma shall contain 19/20 filter points

± 3 Sigma shall contain ALL of the points

If not, corrective action as noted above shall be taken.

Shewhart type control charts may also be used to display and assess the data. These are especially useful for single analyte analyses.

The QA*O* will maintain a list of those methods using control tables or charts.

If required by the project the results of the QC samples may be utilized to calculate and estimation of uncertainty for the reported data in "Estimation of Uncertainty of Measurement (EUM)", MCL-7735.

2.13 Data Review and Evaluation (Design Control)

The PM will define the level of data review required to meet the project quality objectives and customer's expectations. The MCLinc minimum standard for data review is a two level review of an initial (level one) review by the analyst/instrument operator assuring that the analyses was properly performed, calculations are checked and the acceptance criteria for the method were met. The QAO, another analyst or TD will perform a second review (level 2 review) of the parameters listed plus the final report.

During the data review and quality analysis process, any quality data outside of method or project set criteria must be evaluated by the reviewer to determine appropriate corrective action. If the problem is caused by a systematic error, an investigation shall be performed to assure the proper corrective action is established. This approach is necessary to avoid reporting incorrect data to the client.

Additional internal data review (level three) will be provided by appropriate senior technical staff as warranted per the subject matter of the data and/or the requirements of the project. In no instance will data be reported without review. Information reported prior to completion of the review/evaluation process must be clearly identified as "preliminary data".

Computer programs that are used to produce test data or calculate test data must be self-checking or verified per "Verification of Data Software," MCL-7728.

2.14 Standard Operating Procedures (SOPs)

MCLinc uses SOPs to define routine analytical methods, quality systems, chemical hygiene, health and safety, security, and radiological. For simpler routine procedures, QC, or project specific requirements, MCLinc uses operator aids that are incorporated as controlled documents in MCLinc SOP MCL-7756, "Operator Aids," or as an attachment to an SOP.

The key elements of SOPs and Operator Aids are presented in Table 2.14. Any deviations from these elements must be approved by QA.

Current routine procedures to be covered by this document, operator aids or SOPs include the following:

- Reagent water preparation
- Sample receiving
- Balance checks
- Preparation of standards
- Temperature monitoring of ovens and refrigerators
- Calibration of thermometers
- Preventative maintenance
- Calibration of mechanical pipettes
- Checking of hood velocities
- Detection limits studies
- Preventive maintenance
- Inspection of glove boxes
- Assessment of data

A complete list of SOPs and Operator Aids is found on the MCLinc Controlled Document Status List maintained by the Document Control Coordinator (DCC).

Table 2.14 Key Elements of SOPs and Operator Aids

Section	SOP*	Operator Aid**
Title	Cover Usage in a complete statement referencing a regulatory procedure as appropriate	Clear, simple statement
Purpose	Purpose may be detailed	Included in scope
Scope	Covers use and application	Simple description of purpose and scope
Responsibilities	Defines roles of analyst, supervisor, QA and management	Not applicable
Definitions	Provide any definitions unique to SOP	Define unique terms when used
Reagents	Define each with all the details (i.e., chemical name, formula, % purity, manufacture).	Define in procedure- reagents used and source only if unique
Standards	Define each including concentration or purity	Define in procedure
Equipment	Define equipment or instrumentation utilized	State in procedure equipment used but provide details only on specialized equipment
Reagents Preparation	Describe details of preparing reagents listed above	Include for non-routine reagents in the procedure

Section	SOP*	Operator Aid**
Standardization and Calibration	Define process (including initial preparation of standards), standard numbers, levels, acceptance criteria, etc.	Cover in the procedure in stepwise format
Procedure	Stepwise details of the process which in some cases may include the why or background for each step	Simple steps to follow assuming a trained analyst
Safety (including any waste issues)	May be separate section or defined as necessary in SOP	Define in procedure where appropriate
Calculations	Outline information required and formula to use	Define in procedure the formula and its elements
QC	Define the types of QC samples required and the appropriate criteria for evaluating data. Define corrective action	Define in procedure QC samples required, corrective action, and define on prep sheet QC criteria
Documentation	Define preparation sheet or where data should be documented (i.e., notebooks)	Includes preparation sheet or defines what to record in notebook
Pollution Prevention and Waste Management	In all SOPs dealing with chemicals	Not usually needed
References	List applicable documents	List Documents
Other sections or attachments may be required to meet the needs of the SOP usage	Comment – It is also appropriate to have the SOP summarized as an Operator Aid and enclosed as an attachment.	Rarely needed

* These are the requirements for a SOP covering an analytical procedure. Other administrative or policy SOPs may not need all sections listed.

** An Operator Aid for operating equipment may just include a stepwise procedure. This aid is for an analytical procedure.

3.0 PROCUREMENT, SUBCONTRACTING AND DOCUMENTATION

3.1 Procurement

Procurement planning begins with the PM evaluating the needs of the project including the specifications of the required items. These needs are then compared to the approved sources/vendors.

Procurement will then be done through a qualified and established vendor. When a new vendor must be used prior to qualification, the vendor must provide and/or meet any requested technical and operational specifications that may influence quality, safety, and/or environmental concerns. These specifications are reviewed with the QAO and will become part of the final project documentation. The Controller at the direction of the QAO, maintains a list of approved vendors (in the MCLinc purchasing software database).

Using a MCLinc Purchase Order, the PM documents the desired product that meets the project or use required specifications by catalog or identification number. In cases where it is necessary to assure the quality of the product, the specifications required are defined in the purchase order. The purchase order is used to confirm the material or service upon receipt.

Documentation for project related purchases are maintained in the project files and the PM is responsible to assure the specifications of products received meet project needs.

Upon receipt the procured items, they are checked by the PM or his designee, against the ordered item for compliance prior to use. The desired quality will be checked during initial use for critical consumables, supplies and services that affect the quality of MCLinc services. Any identified quality issue must be immediately reported to the QAO for investigation of root cause and determination of corrective action (See Section 2.7, Material and Sample Receipt).

For the purposes of NQA-1 requirements, MCLinc does not purchase materials for direct Nuclear Facility-Related use. All day-to-day procurements are via purchase order and are commercial grade items with specifications clearly defined by the vendor. If a unique

item is required, the PM under the direction of the QAO/TD reviews the design and/or specifications and seeks and evaluates qualified vendors or sources. Any new vendor must be approved by the QAO.

Further details of the procurement process are defined in "Procurement Control," MCL-7727.

3.2 Subcontracting

When MCLinc uses a subcontractor for support services or testing services that it does not provide, or for workload overflow; the client is informed of this approach and a competent pre-approved subcontractor is used. The subcontractor must meet any certifications required by the project, (i.e., AIHA).

The need for subcontract services is identified in the project planning stage and if the services required are not available through a previous approved subcontractor a new subcontractor will be sought. This involves definition of the requirements for the services needed, along with any certifications required and solicitation of the supporting documentation from potential vendors. The PM will review the documentation and make a recommendation to the QAO/TD for final approval.

MCLinc President — Date: 9/1/15

Quality Assurance Officer — Date: 9/1/15

4.0 NON-CONFORMANCES, CORRECTIVE AND PREVENTATIVE ACTIONS

During the course of normal business activities, problems may arise that potentially impact the quality of the work and/or MCLinc's ability to meet our client's requirements. These problems must be reported by the individual identifying the problem in a timely manner to responsible staff (QAO, TD, LM, or MCLinc President).

The problem will then be investigated and appropriate corrective action taken to resolve and eliminate future reoccurrence as required by 10CFR21, "Reporting of Defects and Non-compliance." The goal of each investigation is to determine where possible the root cause or real source of the error or variance. When found this "root cause" must be documented and become part of the lessons learned information passed on to management and staff. The QAO will randomly assess the documentation and implementation of corrective actions on quality related issues. Consideration will be given during the investigation to any preventive actions necessary to avoid future issues (e.g., change SOP or perform process spot check, etc)."

The MCLinc quality program encourages each staff member to be proactive and point out potential problem areas. Management will implement this preventive action with the same priority as any corrective action.

The non-conformance process and Problem/Action Report format is detailed in "Procedure for Reporting Problems, Non-Conformances and Associated Actions, MCL-7722."

5.0 PERSONNEL TRAINING AND QUALIFICATIONS

MCLinc personnel are qualified to perform their job duties based upon a combination of one or more of the following criteria:

- Formal education
- On-the-job training
- Formal training (vendor courses, site and customer-specific training, etc.)

Training is performance based and proof of successful completion and understanding of the material must be demonstrated and documented. The training and qualification needs of the individual MCLinc staff members are determined by either the TD, or LM. For any new procedure the LM and TD will establish training requirements and have the analyst perform a Demonstration of Capability (DOC). This DOC procedure is explained on a DOC form available from the QAO.

5.1 Training

Personnel shall be in compliance with required training. Formal training classes will be used for the majority of the baseline and/or job-specific required training. MCLinc staff/safety meetings will be used to supplement education in safety- and technical-related issues. Off-site training (vendor schools, short courses, seminars, and conferences) can be used for continuing technical and professional training.

Training for procedure or guideline based methods is performed using required reading assignments and topical review in follow-up staff meetings. On-the-job training can be used to supplement any job performance activity. Details of MCLinc's Training Program are located in "MCL Training Program," MCL-7778.

Training records will be maintained for active laboratory personnel. The current training records for each member of the MCLinc staff will be maintained by the DCC.

New employees receive training in the following areas:

- QA/QC
- Chemical Hygiene Plan

- Health and Safety
- Radiochemistry
- Security

The level of this training will be dependent on employee job assignments, but all training will be documented and placed in their training files.

On-going updated training will be provided to all employees as required. Employees are required to read all SOPs issued to them and ask the QAO any questions or clarifications. On-going SOP training will be provided when significant changes are made to the SOP. The MCLinc staff is encouraged to suggest any training needs they have to better perform their jobs. Also, each year during the annual quality assessment, the need for additional training will be reviewed and the effectiveness of current training evaluated.

5.2 Certification of Qualification

In addition to specific training requirements, there are several areas of MCLinc operations that require special/specific qualifications. These are outlined below. If required by the project, this qualification must be further documented and clearly identify the area of qualification and the basis including, as required, any supporting documentation. See example Certificate of Qualification in Appendix E.

5.3 Instrument Operator Qualifications

Operator status will be determined and confirmed by either the TD or the LM. Appendix D lists the instrumentation which requires approval and the MCLinc personnel that are authorized (as of the date shown) as operators. The QAO is responsible for maintaining and distributing updates for the authorized operator listing.

5.4 Radiological Materials

MCLinc staff members must meet the training and authorization requirements as defined by the RSO. The specific requirements for radiological use authorization are defined in the Tennessee Department of Environment and Conservation, Division of Radiological Health, Application for Radioactive Material License and/or the DOE Radiological Protection Program.

5.5 Classified Materials

MCLinc staff members must meet all of the requirements specified in the "Facility Security Plan," MCL-7706. The most important criteria is the need-to-know. This aspect of control over the unauthorized distribution of classified and controlled information will be fully enforced within all aspects of MCLinc business practice. The FSO will maintain all associated documentation and records that are required for compliance with the Facility Security Plan.

6.0 TECHNICAL PROGRAMS

Since the vast majority of MCLinc's technical programs are non-routine or first time research driven activities, the work is performed based on the work plan or scope of work agreed upon with the client. The guidance to execution of their work is found in the MCLinc SOPs including Project Management and Instrument Operational Guides. The MCLinc President and the QAO must approve MCLinc SOPs. All laboratory work is documented in laboratory notebooks to assure recreation of the process followed. The other various guidelines, procedures, and plans that form the basis for the operations and quality performance of MCLinc are listed in the Reference Section of MCLinc's Controlled Documents, Volumes I, II and III.

6.1 Pre-Project Activities

Consideration will be given to the quality, safety and environmental impacts on project performance during the project conception, bidding, procurement, and initiation phases. These areas will be addressed either informally or formally for all MCLinc work. These issues will be documented when dealing with a customer whose work scope is estimated to take more than 80 man-hours to complete. This consideration will help ensure that all customer and MCLinc data quality objectives can be established and met during the successful completion of the work scope.

6.2 Project Conception

Project ideas will be reviewed by MCLinc staff members to determine if the work being requested or proposed is within the capabilities of the MCLinc staff, facility, and resource allocation. Consideration as to resources, facility requirements, waste generation/disposal, and total project life cycle costs and requirements will be considered. Discrepancies or concerns will be presented to either the TD or LM to obtain resolution on the discrepancies or concerns.

6.3 Bidding

When providing a cost estimate or quote for a specific set of services to a customer, the PM will have the cost estimate reviewed by either the MCLinc President, TD, or LM. The internal cost breakdown analysis will demonstrate that consideration has been

provided to meet the customer's quality, documentation, reporting, sample management, procurement, and waste disposal requirements within the cost estimate being provided.

6.4 Project Acceptance

Before a project is accepted and begun, the PM will meet with the LM and TD to make sure that all required MCLinc resources will be available and can be allocated for the successful completion of the customer work package. This includes a review of any possible procurement of goods and services to complete the project.

6.5 Project Documentation and Communication

At the discretion of the PM and the customer, the amount of project specific QA documentation and procedures will be determined. These documents are used as guidance and can be informally approved and accepted between the PM and the customer. The documents will be part of the permanent project file and are the responsibility of the PM to ensure that all proper documentation is archived. The project specific documentation may include but is not limited to:

- QAP,
- Data package/reporting requirements,
- Specific technical procedures or operational methods,
- Enhanced Chain of Custody procedures,
- Calibration and/or certification requirements,
- Environment, health and safety (EH&S) guidance,
- Budget, schedule, and deadline information, and/or
- Correspondence.

A key element of the MCLinc Project is effective communication to the client not only of the project problems or issues, but progress and significant achievements. This communication also allows an opportunity for input to the project from the client on technical matters, opinions and interpretations of the results. In most cases this input is best received during the project than after the fact. The mode of this communication is best dictated by the client and may mean phone calls, meetings, e-mail or other written progress reports. Document all oral communication to assure your understanding of the discussion.

6.6 Reporting and Project Closure

Report structure, detail, organization, and media selection will be determined by the PM and customer. The PM will ensure that all data reviews, data tabulations, laboratory work, and customer requests have been fully completed and documented prior to the compilation of the final report. Any non-conformity with the customer's request will be communicated and documented as soon as possible with the customer. Documented resolution will be noted within the project notes and summary. The PM will ensure that a complete data set,

laboratory notebook reference list, data location listing, and final copy of the customer report are maintained in their files. A copy of the final customer report will be maintained within the MCLinc project files. If a report is to be amended, the report is either reissued or marked as a new revised version, or a clearly defined amendment to the report is issued. In both cases, the new document is sent to the same distribution as the original report.

Upon completion of the project, the PM must place or reference all applicable documents in the project file, make sure any non-routine or special wastes generated during the project are properly stored and/or disposed per "Waste Management Plan," MCL-7718, and that all samples and residues are properly stored for disposal or returned to the client per the contract.

6.7 Clients Complaints

MCLinc welcomes feedback from our clients, be it positive or negative. Despite the efforts to the contrary, the probability exists that the client may express concerns or disfavor with the project to MCLinc staff. Anyone aware of such a complaint must report it to the appropriate QAO, PM or TD for follow-up. It is critical that MCLinc understand completely both sides of the complaint, the root cause and take immediate corrective action. The complaint will be documented with a nonconformance or corrective action memo per MCL-7722, "Procedure for Reporting Preventive Actions, Problems, Non-conformances, and Associated Actions." The QAO will review this corrective action and discuss with client as necessary. Since the majority of our projects include direct contact with the client, discussions concerning their satisfaction or dissatisfaction with our work can be held one on one with any staff member.

7. 0 DOCUMENT CONTROL PROCEDURES

7.1 Document Control

Documents will be managed to ensure that a consistent record of activities exists to allow for a detailed review of current practices to determine if any modifications would permit the improvement of any process, in part or in whole. Documents which are determined to be important to the operation and control of materials and information within MCLinc will be controlled. Controlled documents will be maintained with respect to "Document Control," MCL-7703. Examples of controlled documents are:

- QAPs
- Quality Documents prepared for clients
- Standard Operating Procedures
- Chemical Hygiene Plans
- Health and Safety Plans
- Waste Management Plans
- Operator Aids

During the annual internal audit by the QAO, all controlled documents will be reviewed and if revisions are necessary, they will be scheduled and implemented. Those not revised will be marked "Reviewed without Revision with the date" in the Controlled Document Status form.

7.2 Changes to Controlled Documents

Changes to controlled documents may be initiated by anyone using the document to clarify or correct an error or reflect a change in the procedure. Changes shall be reviewed and approved by the same functions that approved the original document. Information needed to evaluate the requested change, if necessary, should be provided along with the MCLinc Change Form (Example in SOP, "Document Control," MCL-7703). The changes shall be noted on the change form and if required for clarity attachment of the revised document pages. Once signature approval is complete the DCC will issue a controlled copy of the change form and any attachments to all recipients of the original controlled document.

7.3 Notebooks

A critical document in use within MCLinc to record day-to-day work efforts, analyses, and experimentation is the laboratory notebook. Laboratory notebooks are issued by the DCC to individual personnel. These notebooks are assigned with a unique identification number and are maintained by the individual MCLinc personnel. The notebook is the responsibility of the individual user. It is good practice to maintain an index in the front of the book to track the time frames associated with various customers and/or projects which have documentation in the notebook. If it is felt that a section or entry into the logbook should be witnessed, the logbook owner is responsible for providing another cognizant MCLinc staff member to read, verify, and sign the logbook pages that the material has been properly documented and dated. Notebooks, when completed or retired, are returned to the DCC for safe storage. Notebook(s) will be randomly reviewed for compliance to the SOP, "Good Notebook Keeping Practices," MCL-7724, during the year as part of each internal assessment by QA or the TD.

8.0 RECORDS

8.1 QA Records

Records that show or demonstrate evidence of quality or a quality system are deemed quality records. They are to be legible, identifiable and retrievable. QA records may be hard copy or electronic media files. Quality records are maintained by the DCC and the QAO and include the following:

- Current and historical controlled documents
- Laboratory notebooks
- Laboratory / instrument logbooks
- Training files/records
- Instrument output, results, notes, design documents and calculations
- Standards traceability documentation
- Radiochemical inventory documentation (maintained by RSO)
- Non-conformance reports
- Demonstration of Capability Form (DOC)

The PM maintains QA Records that are specific to a project such as standard runs, daily calibrations, calculations and results in the project files.

8.2 Project Records

All technical and business records associated with a project constitute Project Records and are maintained accessible to the project staff during the project and are considered client proprietary. The technical records are maintained by the PM in the designated project files and the business records by the DCC in the Administrative Office files. Examples of Project Records are:

- Work plans or scope of work documents
- Project QAPs
- Project correspondence including phone logs
- Interim and final reports
- Computer files of project information
- Proposals, contracts and change orders

Further details on records are outlined in "Quality Assurance Records,"MCL-7729.

8.3 Record Retention

Record retention is the key to assuring our clients that information if needed in the future is retrievable. Project records are maintained for five (5) years or as otherwise defined in the project contract. QA Records not associated with a project are considered lifetime or permanent records and will be maintained for the usable life of the item. All records are maintained within the secure MCLinc facility in clean, dry areas with access controlled by the The Sample/Report Management Staff (SRM). The SRM receives project documents from the MCLinc staff and places the records into appropriate filing cabinets or new storage boxes and logs the contents into a records storage log which is then used to track the documents for future retrieval. All documents in storage are accessible only through the SRM or QAO.

9.0 ASSESSMENTS

9.1 Management Assessment

Ultimate responsibility for QA/QC and ES&H compliance within MCLinc rests with the MCLinc President. Unresolved MCLinc issues will be resolved by MCLinc management. MCLinc evaluates its performance in January for the previous year with an Annual Management Quality Assessment in January of each year. During this Annual Management Quality Assessment, issues are raised, resolved and documented. The purpose of the Annual Management Quality Assessment is to provide a means to understand the effectiveness of the management system, make recommendations for improvement to top management and implement the improvements. Tools like the quality policy, client and laboratory QA objectives, performance test (PT) sample results and internal and external assessments are used to allow these improvements while maintaining the integrity of the system.

As part of the MCLinc Annual Management Quality Assessment, the MCLinc management review shall take account of:

- Quality objectives of management met
- The suitability of policies and procedures
- Reports from management/supervisory staff
- Results of internal or third party audits/assessments
- Corrective and preventive actions
- The results of round robin or any PT program
- Changes in volume and type of work
- Client feedback or complaints
- Manpower/equipment needs
- Staff recommendations for improvement
- Other relevant factors such as QC activities resources and staff training

Upon completion of the draft Annual Management Quality Assurance Review, the document is submitted to the MCLinc President/Chief Executive Officer (CEO) for review and determination of any findings. Any findings resulting from this management

review will be defined with a designated person responsible and an agreed upon time schedule.

The management assessment will be documented by the QAO.

9.2 Internal Assessments

The QAO on an annual basis will schedule and initiate assessments of the internal quality systems of all the laboratory operations using a total review or checklist approach and documenting all findings in a memo report. These assessments may be performed in part periodically (i.e., monthly) or on a single event. The QAO will define or approve the corrective actions and follow-up as necessary to assure corrective actions have been implemented. A third party independent quality systems assessment sponsored by MCLinc meets the requirement for this assessment. Appendix F is the 2014 MCLinc assessment schedule.

9.3 Independent Assessments

Clients or MCLinc may utilize other organizations, independent of the day-to-day operations of the MCLinc facility, to provide an assessment of quality, safety, and environmental activities within the MCLinc facility. MCLinc will provide a safety orientation to the members of the independent assessment team at the beginning of the assessment kick-off meeting.

All documentation generated by the independent assessment will be addressed in a closeout report that will be generated by the appropriate MCLinc staff no later than twenty-five (25) working days after the independent assessment results are presented to management. Corrective actions will be documented and their effect on the deficiency tracked and noted. If it is felt that the corrective action has had a significant impact on other areas of operation, the corrective action documentation will be used by the appropriate MCLinc staff to compile a positive lesson learned document to ensure that all portions of the MCLinc organization is aware of the potential positive influence of the corrective action. MCLinc may initiate a third party additional audit for specific areas of the laboratory or total laboratory operation.

9.4 Performance Evaluation (PE) and Performance Testing (PT)

MCLinc will participate in PE and PT programs as necessary to evaluate the quality performance of the laboratory. MCLinc currently participates in Mixed Analyte Performance Evaluation Program (MAPEP) (Inorganic and Rad - soil and water); AIHA for metals, air asbestos, beryllium oxide, and bulk asbestos; internal PEs for hexavalent chromium and mercury; and a third-party asbestos program. Others will be added as needed. Any non-passing score in these programs will be investigated and a written report submitted to the QAO within 21 calendar days. *Supplemental PE samples are hex-chrome in water and PCBs in oil, both bi-annual.* The QAO will approve and follow-up on the corrective actions as needed.

10.0 REFERENCES

ANSI/ASQC E4-1994, "Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs."

ASME NQA-1-2000, Edition, "Quality Assurance Program Requirements for Nuclear Facilities."

DOE, "Consolidated Audit Program Quality Systems for Analytical Services Revision 2.9."

Energy, Nuclear Safety Management, Quality Assurance Requirements, Scope, 10 CFR Part 830.120.

Energy Reporting Defects and Noncompliance, 10 CFR Part 21.

ISO/IEC Standard 17025 – "General Requirements for the Competence of Testing and Calibration Laboratories, 2005."

MCL-7702, "Chemical Hygiene Plan."

MCL-7703, "Document Control."

MCL-7704, "Project Management Guide."

MCL-7705, "Nuclear Materials Control and Accountability Plan."

MCL-7706, "Facility Security Plan."

MCL-7708, "Electron Microscopy Operation Guide."

MCL-7710, "Sample Preparation Guide."

MCL-7711, "Calibration, Inspection, and Maintenance Guide."

MCL-7715, "Radiation Protection Plan."

MCL-7717, "Health and Safety Plan."

MCL-7718, "Waste Management Plan."

MCL-7719, "Asbestos Laboratory Quality Assurance Manual."

MCL-7722, "Procedure for Reporting Problems, Non-Conformances, and Associated Actions."

MCL-7724, "Good Notebook Keeping Practices."

MCL-7727, "Procurement Control."

MCL-7728, "Verification of Data Software."

MCL-7729, "Quality Assurance Records."

MCL-7735, "Estimation of Uncertainty of Measurement (EUM)."

MCL-7756, "Operator Aids."

MCLinc's Controlled Documents, Volumes, I, II, and III.
"National Environmental Laboratory Accreditation Conference Standards," Latest Approved Edition

MCLinc President — Date: 9/1/15

Quality Assurance Officer — Date: 9/1/15

Section: Appendices
Section No: 11
Revision: 14
Date: 09/01/2015

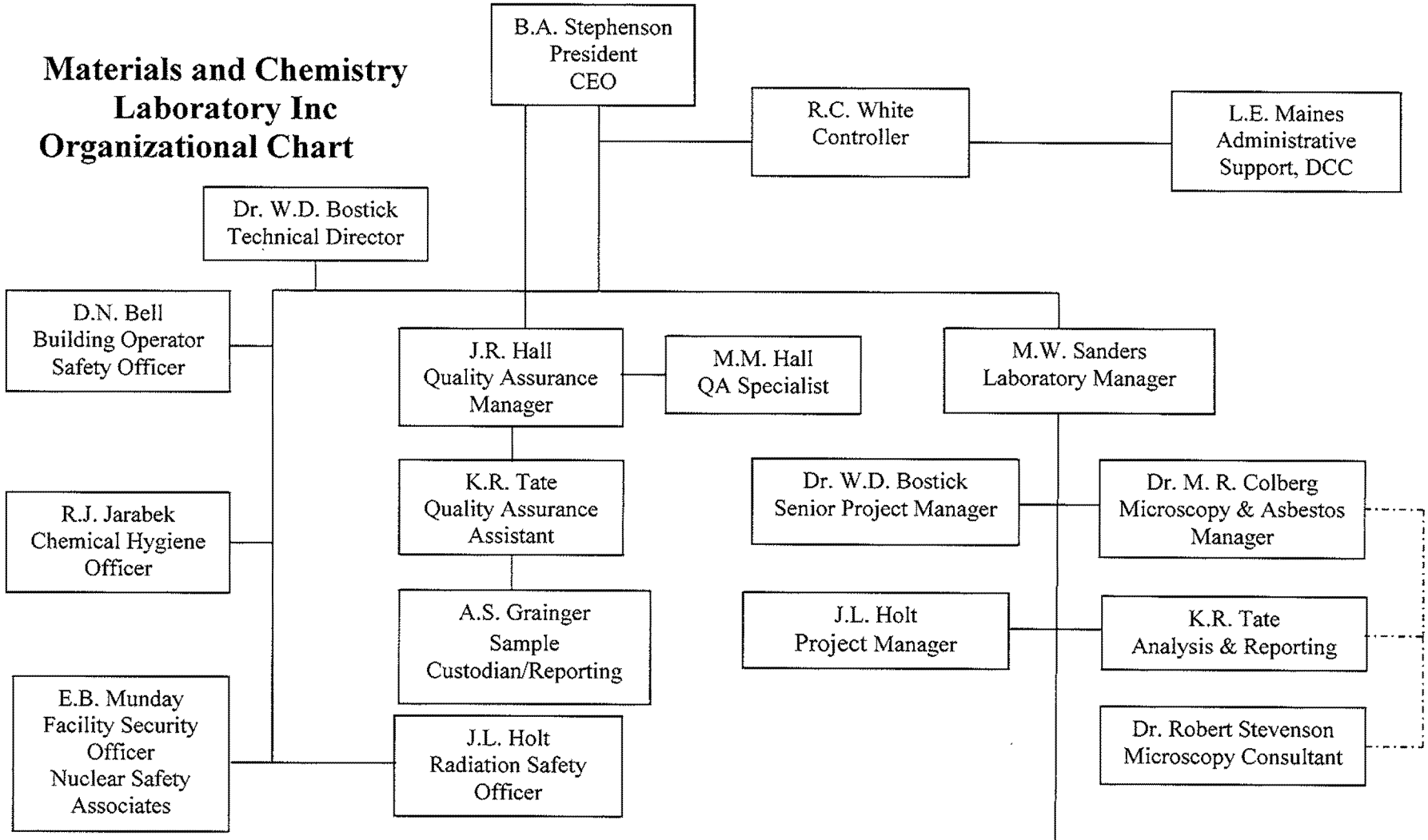
Appendix A - MCLinc QAP Cross-reference to National and International Quality Requirements

Basic Requirements of NQA-1	Requirements of ISO/IEC 17025	MCLinc QAP Section	10 CFR 830.120	ANSI/ASQC E4-1994
Organization	Organization and Management	1.0 Organization	Management	Management and Organization
Quality Assurance Program	Management System Personnel	2.0 Management Systems 5.0 Personnel Training and Qualifications	Quality Assurance Program; Personnel Training and Qualifications	Quality Systems and Description; Personnel Qualifications and Training
Design Control		2.13 Data Review and Evaluation	Design	Design
Procurement Document Control	Review of Requests, Tenders and Contracts	3.0 Procurement, Subcontracting and Documentation	Procurement	Procurement; Planning and Scoping
Instructions, Procedures, and Drawings	Technical Requirements	6.0 Technical Programs	Documents and Records	Documents and Records; Design of Systems
Document Control	Document Control	7.0 Document Control	Documents and Records	Documents and Records
Control of Purchased Items and Services	Purchasing Services and Supplies	3.1 Procurement	Procurement	Procurement
Identification and Control of Items	Measurement Traceability	2.1 Standards and Reference Materials	Procurement	Procurement
Control of Processes	Accommodation and Environmental Conditions	2.0 Quality Systems	Performance-Work Processes	Implement Work Processes; Operation of Systems
Inspection	Subcontracting of Tests and Calibrations	2.12 Data Review 3.2 Subcontracting 9.2 Internal Assessments	Inspection and Acceptance Testing	Computer Hardware/Software; Implementation of Planned Operations
Test Control	Assuring the Quality of Test and Calibration Results	2.2 Calibration	Quality Improvement	Quality Improvement
Control of Measuring and Test Equipment	Test and Calibrations Methods and Method Validation Equipment	2.2 Calibration 2.11 Instrumentation	Quality Assurance Criteria	Quality Systems
------	Service to the Client; Complaints	6.0 Technical Programs	Quality Assurance Criteria	Planning and Scoping
Handling, Storage, and Shipping	Sampling	2.10 Sampling and Sample Preparation	------	Design of Data Collection
Inspection, Test and Operating Status	Handling of Test and Calibration Items	2.0 Quality Systems	Inspection and Acceptance Testing	Design of Data Collection/Verification and Acceptance
Control of Non-Conforming Items	Control of Non-conforming Testing and/or Calibration Work	4.0 Non-conformances, Corrective and Preventative Actions	Quality Improvement	Quality Improvement
Corrective Action	Corrective Action Preventative Action	4.0 Non-conformances, Corrective and Preventative Actions	Quality Improvement	Quality Improvement
Quality Assurance Records	Reporting of Results Control of Records	6.6 Reporting 8.0 Records	Documents and Records	Documents and Records
Audits	Internal Audits Management Reviews	9.1 Management Assessment 9.2 Internal Assessments 9.3 Independent Assessments	Management Assessment; Independent Assessment	Assessment and Response

Appendix A Cross Reference (Continued)

DOE Quality Systems for Analytical Services Latest Revision		MCLinc Quality Assurance Plan Revision 14	
Section	Title	Section	Title
1.0	Introduction, Scope, Applicability	1.1	Introduction
2.0	References	10.0	References
3.0	Terms and Definitions		Defined throughout QAP
4.0	Management Requirements	1.0 2.0	Organization Management Systems
4.1	Organization	1.0	Organization
4.2	Management	2.0	Management Systems
4.3	Document Control	7.1	Document Control
4.4	Review of Requests, Tenders and Contracts	6.3	Bidding
4.5	Subcontracting of Environmental Tests	3.2	Subcontracting
4.6	Purchasing Services and Supplies	3.1	Procurement
4.7	Service to the Client	6.5	Project Documentation and Communication
4.8	Complaints	6.7	Client Complaints
4.9	Control of Nonconforming Environmental Testing Work	4.0	Non-Conformances and Correction Preventative Action
4.10 4.11	Improvement Corrective Action	4.0	Non-Conformances and Correction Preventative Action
4.12	Preventive Action	4.0	Non-Conformances and Correction Preventative Action
4.13	Control of Records	8.0	Records
4.14	Internal Audits	9.2	Internal Assessments
4.16 4.17	Management Reviews Data Integrity Investigations	9.1	Management Assessments
5.0	Technical Requirements	2.0	Management Systems
5.1	General	2.0	Management Systems
5.2	Personnel	5.0	Personnel Training and Qualification
5.3	Accommodation and Environmental Conditions	2.4	Work Environment
5.4	Environmental Test Methods and Method Evaluation	2.14	Standard Operating Procedures
5.5	Calibration Requirements	2.11	Instrumentation and Maintenance
5.6	Measurement Traceability	2.1 2.5	Standards and Reference Materials Laboratory Supplies
5.7-5.8	Sampling and Handling of Samples	2.7 2.8 2.10	Material and Sample Receipt Controlled Samples Sampling and Sample Preparation
5.9	Quality of Environmental Test	2.12 2.13	Quality Control Samples and Assessments of Data Data Review and Evaluation
5.10	Reporting the Results	6.6	Reporting and Project Closure
6.0	Hazardous and Radioactive Materials Management and Health and Safety Practices		Various Locations and Two Separate SOPs (MCL-7718;& MCL-7717; and MCL-7715)
6.1	Radioactive Materials Management and Control	5.4	Radiological Materials and Radiation Protection Plan, MCL 7715 SOP
6.2	TSCA [Toxic Substance Control Act of 1976] Materials		Chemical Hygiene Plan MCL 7702 SOP
6.3	Laboratory Health and Safety		Health and Safety Plan MCL 7717 SOP
6.4	Waste Management and Disposal		Waste Management Plan MCL 7718 SOP

Materials and Chemistry Laboratory Inc Organizational Chart



Appendix C - MCL Support Function Assignments

Position	Personnel
Company Controller	Robert C. White
Chemical Hygiene Officer	Robert J. Jarabek
Classified AIS Security Site Manager	Mark R. Colberg
Classified AIS System Security Officer	Earl B. Munday with Charlie Coffey
Classified Document Custodian	Earl B. Munday
Classified Document Custodian - Alternate	Robert J. Jarabek
Document Control Coordinator	Linda E. Maines
Facility Security Officer	Earl B. Munday
Facility Security Officer - Support	*Atkins Nuclear Solutions US*
MBA Custodian(s)	Robert J. Jarabek Earl B. Munday
NMC&A Manager	Earl B. Munday
NMC&A Alternate Manager	*Mary M. Hall*
OPSEC Manager	Phyllis Ferguson
OPSEC Alternate Manager	Earl B. Munday
MCLinc President	Barry A. Stephenson
Laboratory Manager	Michele W. Sanders
QA Officer	Jack R. Hall
QA Officer-Alternate	Mary M. Hall
Radiological Safety Officer	Jeff L. Holt
Radiological Safety Officer - Alternate	Michele Sanders
Security Container #1 Custodian	Earl B. Munday
Security Container #1 Custodian - Alternate	Mark R. Colberg
Security Container #2 Custodian	Earl B. Munday
Security Container #2 Custodian - Alternate	Robert J. Jarabek
Security Container #3 Custodian	Mark R. Colberg
Security Container #3 Custodian - Alternate	Earl B. Munday
Safety Officer	David N. Bell
Technical Director	William D. Bostick

Appendix D – Instrumentation with Responsible Owner and Authorized Operator

Type	Model	Manufacturer	MCLinc Owner
FTIR	MB100	BOMEN	Munday
FTIR	GL3020/Nicolet (6400)	Mattson	Bostick/Tate
GC (3) 2EC + FID	5890 (2), 7890A	Hewlett Packard	Sanders/ Holt/Tate
IC	ICS1100	Dionex	Sanders/Holt/Dyke/Hensley
ICP	2000	Perkin Elmer	Sanders/Jarabek/Holt/Dyke
ICP/MS	Elan 9000	Perkin Elmer	Sanders/Holt
Mercury Analyzer (AA Cold Vapor)	410	Buck	Sanders/Dyke/Hensley
Mercury Analyzer (Low Level)	Hydro C	Teledyne-Lehman	Holt/Hensley*
Mercury Analyzer (Low Level)	Hydro II AF GOLD	Teledyne-Lehman	Holt/Hensley*
Optical Microscope	Various	Various	Colberg/Jarabek/D. Hall/ Tate/Holt
Rad Spectroscopy	Various	Various	Jarabek/Bostick
SEM	840	JEOL	Colberg/Dunaway*
SEM	ESEM-2020	Philips	Colberg/Dunaway*
SEM	S-4500	Hitachi	Colberg/Dunaway*
SEM	S-5000	Hitachi	Colberg/Dunaway*
TEM	2000FX	JEOL	Colberg/Dunaway*
UV-Vis Spectroscopy	PC1000	Ocean Optics	Bostick
XRD	MiniFlex II	Rigaku	Colberg/Tate
TGA/DTA+MS+GC	6300 ThermoStar GSD301/8610C	SII/Pfeiffer/SRI	Tate/Sanders

*In training

AA-Atomic Absorption
DTA – Date Transfer Analyzer
EC – Electron Capture
FID – Flame Ionization Detector
FTIR – Fourier Transform Infrared Spectroscopy
GC – Gas Chromatography
IC – Ion Chromatography
ICP – Inductively Coupled Plasma
MS – Mass Spectroscopy
SEM – Scanning Electron Microscope
TEM – Transmission Electron Microscope
TGA – Thermogravametric Analysis
UV-Vis – Ultraviolet – Visible
XRD – X-Ray Defraction

Appendix E - CERTIFICATE OF QUALIFICATION AND AUTHORIZATION

MCLinc CERTIFICATE OF QUALIFICATION

Certification of: _____

Certified To Perform: _____

_____

_____

Certification based on:

- ☐ Education
- ☐ Indoctrination
- ☐ Experience
- ☐ Training
- ☐ Test Results (Attach)
- ☐ Capability Demonstration:

  (Observed by: _____)

Certification Level (I, II, III, per NQA-1): _____

Technical Director/QAO Approval: _____

Date of Certification: _____

Expiration Date: _____

Results of Periodic Evaluation: _____

_____

_____

Appendix F

MEMO

DATE: December 30, 2014

TO: Barry A. Stephenson, MCLinc Staff

FROM: Jack R. Hall

FAX#: (865) 576-8558 PHONE: (865) 574-9923

SUBJECT: McLinc QA Internal Assessment Schedule for 2015

MEMO:

As part of the MCLinc QA Plan there is a requirement for an annual assessment, which I am scheduling to occur over the year covering the various quality systems. The schedule is as follows:

January/February	Complete 2014 Management Assessment /Radiation Plan Review-Assessment
March/April	Project files, QA files, and Training files
May/June	Review QAP/ Corrective/Preventative Action Process
July/August	Instrument/Equipment Calibration/Reference Materials + Update QAP
September/October	Sample Log-in Process/ Random Notebook Review
November	Industrial Hygiene Laboratory Internal Assessment per AIHA
December	Procurement and Document Control and perform QA SOPs Review

As part of these quality systems assessments I will be asking for your help to take any needed corrective actions.

Materials and Chemistry Laboratory, Inc.
East Tennessee Technology Park, Building K-1006
2010 Highway 58, Suite 1000, Oak Ridge, Tennessee 37830-1702
Phone: (865) 576-4138 Fax: (865) 576-8558

MATERIALS CHEMISTRY LABORATORY, INC. MCL
STANDARD OPERATING PROCEDURES

MATERIALS AND CHEMISTRY LABORATORY, INC. STANDARD OPERATING PROCEDURE	
Operation Guidance Electron Microscopy: Materials and Chemistry Laboratory, Inc.	Approved: MCLinc President — Date: 2/17/2009 Quality Assurance Officer — Date: 2/16/09

1.0 INTRODUCTION

This document and the documents referenced herein provide a framework for the safe and consistent operation of electron microscopes. It is accepted that operating personnel have an understanding of the instrumentation and theory of operation. This guideline will identify the hazards associated with the operation and ensure the safe usage along with providing a high level of confidence in the results obtained.

2.0 GENERAL RESPONSIBILITIES

2.1 Principle Operator

The Principle Operator is responsible for the routine operation, upkeep of the instrumentation, documentation, and work area associated with the instrumentation. The appointment of the Principle Operator for each instrument is made by the *Chief Operating Officer.*

2.2 Secondary Operator

The Secondary Operator should be able to assist the Primary Operator in routine operation and maintenance. The Secondary Operator may be able to perform all operations at the same level of expertise as the Primary Operator, but this is not a requirement. Secondary Operators may be certified by either the *Chief Operating Officer* or the Principle Operator.

2.3 *Chief Operating Officer*

The *Chief Operating Officer* represents the first level of line management which is responsible for supplying the resources for proper upkeep of the required instrumentation.

3.0 EQUIPMENT AND MATERIALS

3.1 Major Components

This table lists the major equipment covered by this guideline. The property number is the property number associated with the main instrument component. It is recognized that additional property numbers may exist for accessories and other secondary components.

Manufacture	Model #	Property #	Room #
Hitachi	S-4500	K333391	A104
JEOL	JXA-840	K322408	A106
JEOL	2000FX	K331267	E101
Hitachi	S-5000	K333393	E102
FEI	ESEM-2020	K333395	E103

3.2 Basic Process Description

Electron microscopy (EM) impinges a focused electron beam on a solid surface to produce electron *images* and x-rays which contain information about the sample. The electrons are used to create electron micrographs (images) and the x-rays are used to obtain elemental information about the sample *with associated x-ray analyzers*. EMs vary by the nature and relative position of their electron optic components with respect to the sample. EMs can optimize various electron-sample interactions (i.e. scanning, transmission, and diffraction) to obtain various types of materials characterization. The "output" is typically an electron micrograph *from a secondary electron detector (SEI), backscattered electron detector (BEI), or transmitted onto a fluorescent screen*, electron diffractogram, or elemental *composition by x-ray spectroscopy* (qualitative or quantitative). The following are brief overviews of typical operational aspects of the instrumentation:

The electron guns operate at very high voltage (1,000 to 200,000 volts) but at very low current (nA to pA range). EMs operate in a vacuum with the electron gun typically being at 10^{-6} to 10^{-7} torr and the sample being between 50 and 10^{-5} torr; hence, sample exchange and manipulation are done via sample exchange interlocks and mechanical stages.

Each instrument has associated equipment required for the vacuum system, cooling, and valving (compressors). Operators are required to understand the interaction of each component and perform routine, preventive maintenance on each component according to the vendor operating manual.

Each scope has an associated x-ray analyzer used to determine elemental composition. X-ray emission is shielded by the metal construction of each instrument.

3.3 *Basic Operating Process*

This describes the *general* guidelines for *sample preparation, instrument operation, and collecting & transferring data for interpretation.*

3.3.1 *Sample Preparation*

Sample preparation for SEM and TEM investigation is the key for a successful investigation. The following notes should be considered prior to loading a sample into the electron scopes:

SEM: Loose powders are not acceptable in the SEMs. SEM preparations are typically mounted on adhesive carbon tape on top of graphite planchets (ie. Ted Pella, Inc.). Because of sample charging, samples are typically carbon coated to reduce the effect of charging on the images. Feature mapping under a stereoscope prior to analysis is strongly recommended to help navigate on the sample at the higher SEM magnifications.

TEM: Loose powders are not acceptable in the TEM. TEM copper grids with a Formvar film layer can be purchased from Ted Pella, Inc. Three microliter samples can be mounted directly on these grids, dried, and loaded into the TEM. A dispersion in ethanol with gentle sonication works well. The technique for preparing a TEM grid for NIOSH 7402 is outlined in the NIOSH 7402 procedure and MCLinc SOP 7742.

3.3.2 Instrument Operation

Each instrument has a unique start-up/shutdown procedure outlined in each vendor manual. Instruments must be operated according to the vendor operating manual which outlines procedures for loading/unloading samples, operation, data collection, maintenance, and troubleshooting.

Note that the EMs should never be left unattended when the electron source is activated. When not in use, the EMs should be left in the shutdown condition outlined by the principle operator.

3.3.3 SEM Data Collection and Transfer

The SEMs can collect electron images from 20x to 1,000,000x magnification. Both SEI and BEI images can be collected and stored. Images can be transferred for reporting by:

- Polaroid film: Each SEM unit has been set up to collect images by Polaroid type 52 or 57 land film. Film development takes less than 1 minute and has excellent resolution.

- Printer: Each associated x-ray analyzer has the capability to grab the image from the SEM's CRT and print to a printer. The image can then be scanned and converted to a electronic data file.

- Electronic Data File: The Hitachi 4500 has an EDAX x-ray analyzer that is capable of storing and saving images in various formats including bmp, tif, and jpg formats. Data is readily transferred by memory card or CD.

X-ray spectra can be transferred for reporting by:

- Printer: Each associated x-ray analyzer has the capability to print to a printer. The spectra can then be scanned and converted to a electronic data file.

- Electronic Data File: The Hitachi 4500 has an EDAX x-ray analyzer that is capable of storing and saving spectra in various formats including bmp, tif, and jpg formats. Data is readily transferred by memory card or CD.

3.3.4 TEM Data Collection and Transfer

The JEOL 2000FX TEM can collect electron images from 20x to 1,000,000x magnification. Images can be collected and stored only by Kodak film. Follow manufacturer's instructions for using Kodak D-19 Developer and Kodak Rapid Fixer.

X-ray spectra can be transferred for reporting by a printer associated with the x-ray analyzer. The spectra can then be scanned and converted to a electronic data file.

3.4 Laboratory Supplies

This non-inclusive listing provides a baseline for the types of supplies as well as engineering and administrative controls that should be available, as needed, to ensure a safe (personnel and environmental) work place.

Disposable lint-free or powder-free gloves
Lint-free cloths
Disposable laboratory waste bags
Fume hoods equipped to provide a well-ventilated workspace
Protective eyewear
Protective laboratory coat/apron
Spill cleanup material
Emergency eyewash station
Emergency shower station
Fire extinguisher
Access to MSDS sheets for all chemicals used

Ted Pella, Inc and SPI, Inc. are good sources for various EM supplies for sample preparation such as TEM grids and graphite planchets.

3.5 Standards

The following components, or equivalent ones, should be available for quality control and performance evaluation of the various electron microscopes. The selection and use of the particular standard is based upon operator preference. The standard used should be documented in the appropriate logbook and should be used in agreement with the methods outlined in this document.

Magnification standards:

- NIST 484A Specimen ID JY-55-OJ (2 each)
- NIST 484E Specimen ID-SH
- 2160 lines per millimeter cross grating (E. F. Fullam, Inc., Cat. #60021)

Elemental standards:

- C. M. Taylor Corp. #1 Element STD 202-52
- C. M. Taylor Corp. #2 Element STD 202-52
- C. M. Taylor Corp. #4 Element STD 230-27
- C. M. Taylor Corp. #5 Element STD 230-30
- SPI STD 87-103
- Tousimis 8026 103-S

X-ray performance (FWHM) standards:

- X-checker, Small World (#1)
- X-checker, Small World (#2)
- C. M. Taylor Corp. #1 Element STD 202-52

Resolution standards:

- Prickly gold grid Type D

These standards are centrally located, in dry boxes where applicable. Control is maintained through storage in manufacturers labeled containers or in labeled sample storage containers. The standard certification papers are filed with the QA Officer.

4.0 SAFETY PRECAUTIONS

4.1 General Laboratory Safety

Follow guidance outlined in the Chemical Hygiene Plan for the Materials and Chemistry

Laboratory, Inc. (MCL-7702) and the Quality Assurance Plan to the Materials and Chemistry Laboratory, Inc. (MCL-7701).

Develop and encourage safe laboratory habits.
Food will not be stored or consumed in lab areas.
All work areas are to be kept clean and uncluttered.
Safety glasses are required to be worn as posted.
The appropriate personal protective equipment must be worn when required by the job.
Report accidents and near-miss accidents to your supervisor.
On-the-job injuries must be reported immediately.

4.2 Specific Hazards

The electron guns operate at very high voltage (1,000 to 200,000 volts). When changing a filament or performing maintenance, the vendor operating procedure must be followed exactly to prevent high voltage exposure.

For specific hazards of the instruments see the operator's manual and MCL-7717 for Health and Safety approaches to handling the hazards properly. Do not operate unless you understand potential hazards involved with the instrument.

4.3 Emergency Shutdown

The safest, most direct method of shutting the instrument off should be posted in clear plain sight on the front of the instrument. The instructions should be in large print, signed, dated, and laminated.

5.0 ENVIRONMENTAL AND WASTE MANAGEMENT CONCERNS

5.1 Waste Minimization Methods

Kodak Rapid Fixer - Used fixer will be sent out for resource recovery of silver.
Polaroid Film Packs - Digital images will minimize film waste.

Sample Preparation - Use of smallest possible beaker or test tube for cleaning samples or equipment (e.g. tweezers, spatula). Use only a portion of a paper towel or wipe as needed.

Reuse sample planchets by using small amount of double sticky carbon tape. The carbon tape and sample can be peeled off after the analysis and disposed of as solid waste. The planchet can then be reused to mount samples without being added into the waste stream.

5.2 Waste Disposal Methods

All RCRA/TSCA/RAD waste generated by this process shall be disposed of in accordance with the MCLinc Waste Management Plan MCL-7718.

5.3 Environmental Risks

Routine operation of this equipment poses no environmental risks.

6.0 QUALITY AND PERFORMANCE DOCUMENTATION

6.1 Quality Assurance Documentation

The following information should be documented at a minimum of the time period stated and after maintenance activities have been performed. This information will provide direct documentation of the performance (calibration) parameters affecting the quality of the output (results) of the instrumentation. Documentation is the responsibility of the Principle Operator and will be kept with the instrument.

Image magnification (at least semiannually): A determination of the magnification of applicable image source(s) shall be performed.

Energy calibration (at least semiannually): The energy calibration shall be checked. Standards such as Cu and/or Al should be used.

EDS energy resolution - FWHM (at least semiannually): A Mn Ka peak shall be used to measure the full width at half maximum peak intensity (FWHM).

WDS performance (as needed): The position and FWHM of peaks of interest will be documented.

Performance Documentation

The following information shall be documented in the time period stated.

Instrument usage (every time): Logbooks shall be kept for each EM to record instrument usage, operator and project number.

Scheduled instrument maintenance (per event): A copy of the paper work provided by the service provider should be kept in chronological order. Any information or work which has been provided in response to questions or operational abnormalities that is not clearly documented in the paperwork should be documented and attached.

Non-scheduled instrument maintenance (per event): A copy of the paper work provided by the vendor should be kept in chronological order. Any information or system work which is not clearly documented on the vendor's paperwork or work instructions provided over the telephone should be documented.

Instrument calibration non-conformance (per event): The actions required to bring the instrument back into compliance with operating specifications as noted in section 6.1 should be documented.

6.3 Vendor Manuals

Vendor manuals form the basis of documentation for operating information. These manuals in combination with vendor/professional training and on-the-job training should allow the principle operator to safely, properly, and fully operate the instrumentation.

Vendor manuals shall be readily available during instrument operation.

6.4 Data Tracking

Data documentation and archival information is the responsibility of the originator and should be recorded in the laboratory notebook.

7.0 REFERENCES

MCLinc *Chemical Hygiene Plan* (MCL-7702)

MCLinc *Quality Assurance Plan* (MCL-7701)

MCLinc *Waste Management Plan* (MCL-7718)

MATERIALS AND CHEMISTRY LABORATORY, INC. STANDARD OPERATING PROCEDURE	
Operation Guide Sample Preparation: Materials and Chemistry Laboratory, Inc.	Approved: MCLinc President — Date: 4/7/10 Quality Assurance Officer — Date: 3/22/10

1.0 PURPOSE

This document and the documents referenced herein provide a framework for safe and consistent sample preparation. It is assumed that operating personnel have a basic understanding of the sample preparation methods. This guideline will identify the hazards associated with sample preparation and ensure the safe usage of the instruments and methods along with providing a high level of confidence in the results obtained and hence provide the foundation for a quality control and quality assurance program.

It is noted that this plan does not specifically address all aspects of the Asbestos Analysis Laboratory (AAL). The AAL is an American Industrial Hygiene Association (AIHA) accredited laboratory. The AAL operates under a stand-alone quality plan, MCLinc *Industrial Hygiene Laboratory Quality Assurance Manual* (MCL-7719).

This SOP provides general guidelines to sample preparation where as specific sample preparation details may be found in the SOPs appropriate for the analysis.

2.0 ROLES

2.1 Technical Staff

The MCLinc member performing any of the various methods of sample preparation.

2.2 Operations Manager

The Operations Manager represents the first level of line management which is responsible for supplying the resources for proper upkeep of the required equipment.

3.0 EQUIPMENT AND MATERIALS

3.1 Major Components

The use, application, calibration and maintenance of various laboratory components are documented in supplemental QA documents. The proper use and documentation of performance of any equipment used for sample preparation is the responsibility of the individual user.

3.2 Basic Process Description

Sample preparation can be the most important part of an analysis. Yet at the same time it is the most free-style activity of the analysis. Sample preparation utilizes various tools to optimize often the desired characteristic of the sample for the desired analysis. Examples of this include but are not limited to:

- Dispersion of sample for particle size analysis.
- Coating of sample to improve the charge transfer capabilities of the sample.
- Thin layering of a sample to provide bulk analysis but still obtain proper coating.
- Degrease the surface to optimize the sample surface exposure.
- Dry, grind, sieve, size separation, or other physical alterations.
- Prepare epoxy mounts for viewing and analysis or incorporate sample into epoxy mount for viewing and analysis.
- Density and/or size separation and selective sub-sampling.
- Weigh, measure or sub-sample to obtain a known representative working sample.

The many options that exist for sample preparation are often developed over years of trial and error and are best suited to the individual preference. Thus, when performing sample preparation it is important to be aware of the hazards associated with the specific chemicals that are being used as well as the instrumentation and tools being used. It is important to document the methods used for sample preparation to optimize the process (make sure that inappropriate methods are not repeated) and make sure that good methods can be repeated if necessary. Sometimes it is the exact combination of sample and method that works and hence is not reproducible for other samples. It is important that new methods of sample preparation are not performed during off-shift hours.

3.3 Laboratory Supplies

This non-inclusive listing provides a baseline for the types of supplies as well as engineering and administrative controls that should be available to ensure a safe (personnel and environmental) work place.

- Disposable gloves
- Disposable laboratory waste bags
- Fume hoods equipped to provide a well-ventilated work space
- Protective eye wear
- Protective laboratory coat/apron
- Spill cleanup material
- Emergency eyewash station
- Emergency care (911, PSS 574-3282)
- Emergency shower station
- Fire extinguisher
- Access to MSDS sheets for all chemicals used

4.0 SAFETY PRECAUTIONS

4.1 General Laboratory Safety

- Abide by all guidance outlined in the Chemical Hygiene Plan (MCL-7702) and the Quality Assurance Plan (MCL-7701).
- Develop and encourage safe laboratory habits.
- Food will not be stored or consumed in lab areas.
- All work areas are to be kept clean and uncluttered.

- Safety glasses are required to be worn as posted.
- The appropriate personal protective equipment must be worn when required by the job.
- Report all accidents and near-miss accidents to your supervisor.
- All on-the-job injuries must be reported immediately to your supervisor.

4.2 Specific Hazards

These hazards have been identified by the MCLinc Hazard Matrix (see Attachment I). The following sections will list the hazard, its description, what the possible consequences are, and what controls are in place to mitigate the hazard. It is believed that the controls in place are adequate for all hazards identified in this section.

4.2.1 High Noise Level

This hazard is present when samples must be cut down to a smaller size. Cut-off saws used in this preparation can cause a high noise level. Potential consequences are hearing loss in personnel exposed to it. This hazard is controlled by personnel protective equipment, in the correct use of earplugs.

4.2.2 High Temperature

This hazard is associated with ovens and furnaces that may be used in sample preparation. The potential consequences are injury (burns) to personnel from contact with thermal components. The controls to mitigate this hazard include the guidance provided in the vendor manuals, the design of the equipment, and the method (experimental and instrumental design) by which the oven is used. The vendor manuals provide guidance for the proper usage and identification of hazards associated with this equipment. Insulated exterior surfaces and adequate sample transfer areas ensure no contact with hot surfaces occurs. Administrative controls include the labeling of furnaces as HOT and guidance provided in the vendor manuals and in the appropriate references in this document. PPE of thermal gloves, tongs, forceps, and the use of transfer vessels assist in handling hot materials.

4.2.3 Low Temperature

Exposure to low temperatures can occur during the transfer of liquid nitrogen from the transfer dewar to the cold stage or sample preparation. Potential consequences are frostbite or frostburn to personnel from direct contact liquid. This hazard is controlled by both administrative controls and personnel protective equipment (PPE). The administrative control is a guideline that provides the safety guidance and PPE requirements for the transfer of the liquid nitrogen. The PPE required for the transfer (face shield and cryo apron) provides adequate protection from the liquid nitrogen splash that may occur.

4.2.4 High Voltage

This hazard is common for some of the sample preparation equipment (although the majority of it is 110 volt). High voltage is required for instrument operations. Exposure to a high voltage source could lead to electric shock of personnel, destruction of equipment, and possible fire. This hazard is controlled by engineering and administrative controls. Engineering controls exist since these instruments have been manufactured to meet all safety and electrical codes. These instruments provide various safety interlocks to ensure that all sources of high voltage are properly shielded and that unintentional contact with high voltage sources is not possible. Administrative controls exist since maintenance of the equipment is covered by the vendor maintenance contracts. This provides specialized personnel to perform necessary maintenance. It is recognized that the highest risk from the high voltage can occur during non-routine operational conditions. These conditions are when a water leak is present at or near the instrument. Sources of water leaks can be water cooling lines for instrument or from drainage from the piping located above the ceiling panel in the room. Whenever uncontained water is detected, in conjunction with instrument operations, all operations should cease. This off-normal incident should be reported to the MCLinc Operations Manager.

4.2.5 High Pressure

High-pressure gas cylinders are used to provide valve control and support instrumental operations. Although the pressure used by the instrument is not high pressure (<150 psi) the gas cylinder itself represents a high pressure source. Failure of the gas cylinder could lead to asphyxiation or physical injury. This hazard is mitigated by the use of engineering, personnel protective equipment, and administrative controls. The engineering control in place includes a gas transfer buggy that has been designed to transport cylinders and a secure strapping device to keep the cylinder firmly in place after it has been unloaded from the dolly. The PPE includes the use of safety glasses in the case of a high-pressure release or component failure. The administrative control is the MCLinc policy for handling compressed gases. Reference Chemical Hygiene Plan for MCLinc (MCL-7702)

4.2.6 Off-shift Operations

Laboratory work being performed outside of normal shift may create a situation where backup support or help is not immediately available. This may lead to a lack or delay of emergency notification in case of an accident. This hazard is controlled by engineering controls and administrative controls. Engineering controls such as emergency pull boxes, telephones, fire sprinkler system, fire extinguisher and building public address system can be used to notify others of an off-normal situation. Administrative controls exist in that personnel are required to notify the PSS office (574-3282) if they will be occupying laboratory or office facilities during off-shift hours. This notification will help support the emergency response personnel in the event of an off-normal event. The performance of new (i.e. first time) activities are not permitted during off-shift hours. These controls and the use of training and on-the-job experience mitigate the hazards associated with this scenario.

4.2.7 Radioactive Materials

This hazard is common for sample preparation of radiological samples. Handling radioactive materials could lead to personnel exposure and/or contamination of equipment or property. This hazard is controlled by engineering, administrative and PPE controls. The engineering controls are the methods by which the samples are prepared. (All radioactive samples are prepared in a radiological area, in a hood, and are surveyed prior to removal from the area.) For proper analysis the sample has to be stable and firmly in place. The administrative controls are enacted by the HP organization.

4.2.8 Carcinogens

This hazard is common in the preparation of metallography samples. Handling of epoxy/hardeners in metallography samples could expose personnel to carcinogen vapors. This hazard is controlled by both engineering controls and administrative controls. The engineering controls are lab hoods for preparation. The administrative controls are small quantities.

This hazard could expose personnel to microscopic dust in the preparation of samples. High levels of microscopic dust could cause respiratory tract problems in personnel. This hazard is controlled by both engineering controls and personnel protective equipment (PPE). The engineering controls are lab hoods and the PPE controls are respirators.

4.2.9 Flammability

This hazard is associated with several of the chemicals (e.g. acetone, methanol, ethanol) that may be used during sample preparation. This hazard may cause a flash or burn to personnel and/or cause facility/equipment to ignite. The hazards associated with this are mitigated through the combination of engineering, administrative, and PPE controls. The engineering controls include lab hoods, secondary containment, small volume plastic squeeze bottles, fire extinguisher, and fire emergency pull boxes. All of these controls help to reduce the initial occurrence of a flammable incident as well as the spread of fire should a small laboratory fire occur. The administrative guidelines include MCLinc procedures/guidelines/and the use of small quantities of materials. Reference Chemical Hygiene Plan for MCLinc (MCL-7702). All of these controls help to reduce the possibility of an initial occurrence and severity of any occurrence that might happen. PPE controls to mitigate the hazard include the use of gloves and safety glasses.

4.2.10 Combustibility

This hazard is associated with several of the chemicals: (e.g. acetone, methanol, ethanol etc.) that may be used during sample preparation. These chemicals/vapors can combust when used around open flames or sparks. The hazards associated with these are mitigated through the combination of engineering and administrative controls. The engineering controls include lab hoods and strong ventilation of labs. The administrative control is the use of small volume of these chemicals.

4.2.11 Oxidizing or Reducing Ability

Description: corrosivity and adverse reactions. Oxidants may release oxygen and reductants may release hydrogen gas. Strong oxidizers (e.g., nitrate or perchlorate salts) can produce high gas pressure when heated, and may promote or support exothermic reactions or fires. Some compounds are shock-sensitive or explosive, and chemical incompatibilities may result in severe adverse reactions (e.g., mixing concentrated nitric acid with many organic compounds may cause a delayed explosion). Engineering controls are similar to those for flammability hazards. Administrative controls include familiarity with MSDS precautions for storage or use, and material substitution (e.g., clean glassware with detergent solutions, not chromic acid, etc.)

4.2.12 Acidity or Causticity

This hazard is associated with the use of acids or bases in the preparation of samples. This hazard could expose personnel to extreme pH materials that can cause burns and/or irritation. To mitigate this hazard engineering, administrative, and PPE controls are used. The engineering controls used include laboratory hoods, secondary containment, and laboratory clamps to hold the receiving vessel. These controls will help to minimize the chance of a spill and to contain any spill that may occur without exposing personnel. The administrative guidelines include the

use of MCLinc guidance and technical literature on the safe handling of chemicals. The Chemical Hygiene Plan for MCLinc (MCL-7702) helps to educate personnel as to the dangers associated with these materials. PPE controls include the use of safety glasses, tongs, face shields, rubber gloves, lab coats, and rubber aprons. These PPE controls help to prevent the exposure of personnel in the case of an accidental spill or splash.

4.2.13 Toxicity

This hazard is associated with the use of toxic materials in the preparation of samples. This hazard could expose personnel to toxic materials above accepted threshold values. To mitigate this hazard engineering, administrative, and PPE controls are used. The engineering controls used include laboratory hoods, secondary containment, and laboratory clamps to hold the receiving vessel. These controls will help to minimize the chance of a spill and to contain any spill that may occur without exposing personnel. The administrative guidelines include the use of MCLinc guidance and technical literature on the safe handling of chemicals. The Chemical Hygiene Plan for MCLinc (MCL-7702) helps to educate personnel as to the dangers associated with these materials. PPE controls include the use of safety glasses, face shields, rubber gloves, lab coats, and rubber aprons. These PPE controls help to prevent the exposure of personnel in the case of an accidental spill or splash.

4.3 Classified Work

Follow all guidance provided in the MCLinc Facility Security Plan (MCL-7706) for performing classified work.

5.0 ENVIRONMENTAL AND WASTE MANAGEMENT CONCERNS

5.1 Waste Minimization Methods

- Proper design.
- Start small--scale up if needed.
- Sample preparation - use of smallest possible beaker or test tube for cleaning samples or equipment (e.g. tweezers, spatula). Use only a portion of a paper towel or wipe as needed.

5.2 Waste Disposal Methods

All RCRA/TSCA/RAD waste generated shall be disposed of in accordance with MCLinc policies.

5.3 Environmental Risks

All chemicals are used in either small volumes, secondary containment, or closed systems for any open processes. No significant risks exist for the sample preparation activities performed by the MCLinc.

6.0 QUALITY AND PERFORMANCE DOCUMENTATION

6.1 Quality Assurance Documentation

The method used to prepare a sample should be documented in the technical staff member's laboratory notebook. Documentation of the sample preparation methods will meet MCLinc and/or customer quality

7.0 REFERENCES

MCLinc *Industrial Hygiene Laboratory Quality Assurance Manual* (MCL-7719)

MCLinc *Chemical Hygiene Plan* (MCL-7702)

MCLinc *Facility Security Plan* (MCL-7706)

Appendix I - Hazard Evaluation

MCLinc Hazard Evaluation		
ACTIVITY: SAMPLE PREPARATION (Non-asbestos) **LOCATION:** Rms. A102,A104,A106,B104,D101,D103,D105,D109,E100,E101,E102, E103,E104,E105,E108 **EVALUATOR:** D.P. Hoffmann **DATE OF EVAL.:** April 23, 1998		
Potential Hazard	**Exist?**	**Description/Mitigation**
High Noise Level	YES	**DESCRIPTION:** Cut-off saws used in sample preparation can cause a high noise level. **POTENTIAL CONSEQUENCES:** Personnel exposed to high noise levels can cause hearing loss. **CONTROLS TO MITIGATE HAZARD:** Personnel protective equipment (e.g. ear plugs). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
High Temp. (≥250°C)	YES	**DESCRIPTION:** Ovens and/or muffle furnaces used to prepare samples may produce high temperature material handling hazards. **POTENTIAL CONSEQUENCES:** Burn to personnel from handling hot materials. **CONTROLS TO MITIGATE HAZARD:** Engineering controls (e.g. oven design, temperature read-out), administrative controls (e.g. signs, guidelines, vendor manuals), and personnel protective equipment (e.g. gloves, tongs, hot pads).

MCLinc Hazard Evaluation		
ACTIVITY: SAMPLE PREPARATION (Non-asbestos) **LOCATION:** Rms. A102,A104,A106,B104,D101,D103,D105,D109,E100,E101,E102, E103,E104,E105,E108 **EVALUATOR:** D.P. Hoffmann **DATE OF EVAL.:** April 23, 1998		
Potential Hazard	**Exist?**	**Description/Mitigation**
		ADEQUACY OF CONTROLS: 1 - the controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Low Temp.	YES	**DESCRIPTION:** Liquid Nitrogen (LN) is a cryogenic material required for operation of instruments. **POTENTIAL CONSEQUENCES:** Exposure of bare skin to (LN) can cause serious frostbite and freeze burns. **CONTROLS TO MITIGATE HAZARD:** Engineering controls (e.g. Transfer dewar), personnel protective equipment (e.g. full-face shield, insulated gloves, cryo-apron). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
High Voltage (≥220V)	YES	**DESCRIPTION:** Exposure to high voltage source required for operation of instrument. **POTENTIAL CONSEQUENCES:** Electrical shock, burn or electrocution of personnel. Damage to equipment or facilities due to electrical discharge.

MCLinc Hazard Evaluation		
ACTIVITY: SAMPLE PREPARATION (Non-asbestos) **LOCATION:** Rms. A102,A104,A106,B104,D101,D103,D105,D109,E100,E101,E102, E103,E104,E105,E108 **EVALUATOR:** D.P. Hoffmann **DATE OF EVAL.:** April 23, 1998		
Potential Hazard	**Exist?**	**Description/Mitigation**
		CONTROLS TO MITIGATE HAZARD: Engineering controls (e.g. instrument design, safety interlocks), administrative controls (e.g. signs, guidelines, vendor manuals, lock out/tag out). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
High Pressure (≥150 psi)	YES	**DESCRIPTION:** Catastrophic rupture (most likely to valve stem) of compressed gas cylinders. **POTENTIAL CONSEQUENCES:** Rapid release of compressed gas could cause an asphyxiating environment or projectiles/fragments from rupture could injure personnel. **CONTROLS TO MITIGATE HAZARD:** Engineering controls (e.g. gas moving cart, tie down straps), administrative controls (guidelines, MCL/MRI procedures). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Electromagnetic	NO	N/A
Moving Heavy Items	NO	N/A

MCLinc Hazard Evaluation

ACTIVITY: SAMPLE PREPARATION (Non-asbestos)
LOCATION: Rms. A102,A104,A106,B104,D101,D103,D105,D109,E100,E101,E102, E103,E104,E105,E108
EVALUATOR: D.P. Hoffmann
DATE OF EVAL.: April 23, 1998

Potential Hazard	Exist?	Description/Mitigation
Machine Guarding	NO	N/A
Off-shift Operations	YES	**DESCRIPTION:** Lack of emergency notification support in the case of an accident. **POTENTIAL CONSEQUENCES:** Personnel or emergency situations may not be found/noticed for extended periods of times which may intensify the extent of damage. **CONTROLS TO MITIGATE HAZARD:** Engineering controls (e.g. emergency pull boxes, telephone, fire sprinkler system), administrative controls (e.g. guidelines, MCLinc policy). **ADEQUACY OF CONTROLS:** 1 - controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Radioactive Materials	YES	**DESCRIPTION:** Handling of radioactive materials during sample preparation may expose personnel to radiation above ALARA levels. **POTENTIAL CONSEQUENCES:** Personnel may receive exposure to radiation greater than permissible levels. **CONTROLS TO MITIGATE HAZARD:** Engineering controls (e.g. HEPA filtered hoods, frisking stations, radiological control areas), administrative controls (e.g. guidelines, MCLinc HP policies, Bioassay program, handle only low-level materials), personnel protective equipment

MCLinc Hazard Evaluation		
ACTIVITY:	SAMPLE PREPARATION (Non-asbestos)	
LOCATION:	Rms. A102,A104,A106,B104,D101,D103,D105,D109,E100,E101,E102, E103,E104,E105,E108	
EVALUATOR:	D.P. Hoffmann	
DATE OF EVAL.:	April 23, 1998	
Potential Hazard	**Exist?**	**Description/Mitigation**
		(e.g. lab coats, shoe scuffs, gloves, safety glasses). **ADEQUACY OF CONTROLS:** 1- controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Confined Space	NO	N/A
Elevated Working Surface	NO	N/A
Hoisting and Rigging	NO	N/A
Carcinogens	YES	**DESCRIPTION:** Exposure to carcinogen vapors. **POTENTIAL CONSEQUENCES:** Handling of epoxy/hardeners during metallography sample preparation may expose personnel to vapors. **CONTROLS TO MITIGATE HAZARD:** Engineering controls (e g. lab hoods), administrative controls (small quantities). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Fibrous Materials	YES	**DESCRIPTION:** Handling of fibrous materials during

MCLinc Hazard Evaluation		
ACTIVITY:	SAMPLE PREPARATION (Non-asbestos)	
LOCATION:	Rms. A102,A104,A106,B104,D101,D103,D105,D109,E100,E101,E102, E103,E104,E105,E108	
EVALUATOR:	D.P. Hoffmann	
DATE OF EVAL.:	April 23, 1998	
Potential Hazard	**Exist?**	**Description/Mitigation**
		sample preparation could expose personnel to microscopic dust. **POTENTIAL CONSEQUENCES:** Personnel may receive high levels of dust. **CONTROLS TO MITIGATE HAZARD:** Engineering controls (e.g. lab hoods, respirators). **ADEQUACY OF CONTROLS:** 1 - controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Explosive Mixtures	NO	N/A
Glove box operations	NO	N/A
Ability to self-polymerize	NO	N/A
Shock sensitivity	NO	N/A
Thermal instability	NO	N/A
Rearranging Ability	NO	N/A
Pyrophoricity	NO	N/A
Flammability	YES	**DESCRIPTION:** Fire or flash from flammable materials. **POTENTIAL CONSEQUENCES:** Personnel burns or ignition of facility/equipment.

MCLinc Hazard Evaluation		
ACTIVITY: SAMPLE PREPARATION (Non-asbestos) **LOCATION:** Rms. A102,A104,A106,B104,D101,D103,D105,D109,E100,E101,E102, E103,E104,E105,E108 **EVALUATOR:** D.P. Hoffmann **DATE OF EVAL.:** April 23, 1998		
Potential Hazard	**Exist?**	**Description/Mitigation**
		CONTROLS TO MITIGATE HAZARD: Engineering controls (e.g. lab hoods, plastic squeeze bottles, fire extinguisher, fire protection systems, fire emergency pull boxes), administrative controls (e.g. guidelines, no open flames, small quantities), personnel protective equipment (gloves, safety glasses, lab coats). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Combustibility	YES	**DESCRIPTION:** Combustibility from flammable materials. **POTENTIAL CONSEQUENCES:** Personnel injury or destruction of facility/ equipment. **CONTROLS TO MITIGATE HAZARD:** Engineering controls (e.g. lab hoods), administrative controls (no open flames, small quantities, safety team), personnel protective equipment (shields, safety glasses, gloves). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.

MCLinc Hazard Evaluation

ACTIVITY: SAMPLE PREPARATION (Non-asbestos)
LOCATION: Rms. A102,A104,A106,B104,D101,D103,D105,D109,E100,E101,E102, E103,E104,E105,E108
EVALUATOR: D.P. Hoffmann
DATE OF EVAL.: April 23, 1998

Potential Hazard	Exist?	Description/Mitigation
Peroxidizing Ability	NO	N/A
Water reactivity	NO	N/A
Oxidizing or reducing ability	YES	**DESCRIPTION:** Corrosivity and adverse reactions. Oxidants may release oxygen and reductants may release hydrogen gas. **POTENTIAL CONSEQUENCES:** Equipment corrosion or heat/pressure damage; personnel injury (burns or injuries from projected articles). **CONTROLS TO MITIGATE HAZARD:** Engineering controls are similar to those for flammability hazards. Administrative controls include familiarity with MSDS precautions for storage or use, and material substitution (e.g., clean glassware with detergent solutions, not chromic acid, etc.). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Acidity or causticity	YES	**DESCRIPTION:** Exposure to materials with high or low pH. **POTENTIAL CONSEQUENCES:** Chemical burn or skin irritation to personnel. **CONTROLS TO MITIGATE HAZARD:** Engineering

MCLinc Hazard Evaluation

ACTIVITY: SAMPLE PREPARATION (Non-asbestos)
LOCATION: Rms. A102,A104,A106,B104,D101,D103,D105,D109,E100,E101,E102, E103,E104,E105,E108
EVALUATOR: D.P. Hoffmann
DATE OF EVAL.: April 23, 1998

Potential Hazard	Exist?	Description/Mitigation
		controls (e.g. lab hood, vessel clamping/holding systems), administrative controls (e.g. guidelines, technical literature), personnel protective equipment (e.g. gloves, face shield, tongs, safety glasses, rubber apron). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate. **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Toxicity	YES	**DESCRIPTION:** Exposure to toxic materials. **POTENTIAL CONSEQUENCES:** Chemical exposure of personnel to toxic materials. **CONTROLS TO MITIGATE HAZARD:** Engineering controls (e.g. lab hood, vessel clamping/holding systems), administrative controls (e.g. guidelines, technical literature), personnel protective equipment (e.g. gloves, face shield, safety glasses, rubber apron). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate. **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Increased reactivity	NO	N/A

MCLinc Hazard Evaluation

ACTIVITY: SAMPLE PREPARATION (Non-asbestos)
LOCATION: Rms. A102,A104,A106,B104,D101,D103,D105,D109,E100,E101,E102, E103,E104,E105,E108
EVALUATOR: D.P. Hoffmann
DATE OF EVAL.: April 23, 1998

Potential Hazard	Exist?	Description/Mitigation
Ionizing Radiation	NO	N/A

Materials and Chemistry Laboratory, Inc. Standard Operating Procedure	
Operation Guide X-Ray Diffraction: Materials and Chemistry Laboratory, Inc.:	Approved: MCLinc President — Date: 2/24/09 Quality Assurance Officer — Date: 2/24/09

1.0 PURPOSE

This document and the documents referenced herein provide a framework for the safe and consistent operation of the x-ray diffractometer (XRD). It is accepted that operating personnel have an understanding of the instrumentation and theory of operation. This guideline will identify the hazards associated with the operation and ensure the safe usage of the instrumentation. This guideline will provide a high level of confidence in the results obtained and provide the foundation for quality control and quality assurance.

2.0 ROLES AND REFERENCES

2.1 Responsibilities

2.1.1 Principle Operator

The principle operator is responsible for the routine operation, upkeep of the instrumentation, and work area associated with the instrumentation. The appointment of the principle operator for each instrument is made by the *Chief Operating Officer*.

2.1.2 Secondary Operator

The secondary operator should be able to assist the primary operator in routine operation and maintenance. The secondary operator may be able to perform all operations at the same level of expertise as the primary operator, but this is not a requirement. Secondary operators may be certified by either the *Chief Operating Officer* or the principle operator.

2.1.3 *Chief Operating Officer*

The *Chief Operating Officer* represents the first level of line management which is responsible for supplying the resources for proper upkeep of the required instrumentation.

3.0 EQUIPMENT AND MATERIALS

3.1. Major Components

This table lists the major equipment covered by this guideline. The property number is the property number associated with the main instrument component. It is recognized that additional property numbers may exist for accessories and other secondary components.

Manufacture	Model #	Property #	Room #	Principle Operator	Secondary Operator
Philips Electronics	XRG-3100	K308903	D103	*M.R. Colberg*	*K.R. Tate*

3.2. Basic Process Description

X-ray diffraction measures the intensity of x-rays (i.e. Cu Kα) that diffract off a powder sample at discrete angles. The relative angle-intensity relationship provides crystallographic information about the sample. The diffraction pattern serves as a "fingerprint" of the phases of crystalline species present. The following is a brief overview of typical operational aspects of the instrumentation:

- The water cooled x-ray tube operates at high power (2200 watts maximum). Typical operating conditions are 40 kV and 35 mA (e.g. 1400 watts).
- XRD operates at atmospheric conditions.
- X-ray yield is contained/shielded by the instrument.

3.3. Laboratory Supplies

This non-inclusive listing provides a baseline for the types of supplies as well as engineering and administrative controls that should be available, as needed, to ensure a safe (personnel and environmental) work place.

- Disposable gloves
- Disposable laboratory waste bags
- Protective eye wear (during maintenance)
- Spill cleanup material
- Emergency eyewash station
- Emergency shower station

- Fire extinguisher
- Access to MSDS sheets for all chemicals used

3.4 Standards

The following component should be available for quality control and performance evaluation of XRD. The selection and use of this particular standard is based upon operator preference. The standard used should be documented in the appropriate logbook.

- Position/Intensity/Resolution Standard:
- Quartz (Supplied by Philips Electronic Instruments)

4.0 SAFETY PRECAUTIONS

4.1 General Laboratory Safety

- Abide all guidance outlined in the Chemical Hygiene Plan (MCL-CHP-001) and the Quality Assurance Plan (MCL-QAP-001).
- Develop and encourage safe laboratory habits.
- Food will not be stored or consumed in lab areas.
- All work areas are to be kept clean and uncluttered.
- Safety glasses are required to be worn as posted.
- The appropriate personal protective equipment must be worn when required by the job.
- Report all accidents and near-miss accidents to your supervisor.
- All on-the-job injuries must be reported immediately.

4.2 Specific Hazards

These hazards have been identified by the MCLinc. The following sections will list the hazard, its description, what the possible consequences are, and what controls are in place to mitigate the hazard. It is believed that the controls in place are adequate for all hazards identified in this section.

4.2.1 High Voltage

High voltage is required for instrument operations. Exposure to a high voltage source could lead to electric shock of personnel, destruction of equipment, and possible fire. This hazard is controlled by engineering and administrative controls. Engineering controls exist since these instruments have been manufactured to meet all safety and electrical codes. These instruments provide various safety interlocks to ensure that all sources of high voltage are properly shielded and that unintentional contact with high voltage sources is not possible. Administrative control exists since maintenance of the equipment is covered by maintenance agreements with the vendor. This provides highly specialized and skilled personnel to perform all necessary maintenance. These maintenance sub-contractors are also monitored and made to comply with all MCLinc safety rules and regulations. It is recognized that the highest risk from the high voltage can occur during non-routine operational conditions. These conditions are when a water leak is present at or near the instrument. Sources of water leaks can be water-cooling lines for the x-ray source or from drainage from the piping located above the ceiling panel in the room. Whenever uncontained water is detected all operations should cease. This off-normal incident should be reported to the MCLinc Manager.

4.2.2 Off-shift Operations

Laboratory work being performed outside of normal shift may create a situation where backup support or help is not immediately available. This may lead to a lack or delay of emergency notification in case of an accident. This hazard is controlled by engineering controls and administrative controls. Engineering controls such as emergency pull boxes, telephones, fire sprinkler system, fire extinguisher, building public address system can be used to notify others that of an off-normal situation. Administrative controls exist in that personnel are required to notify the PSS office (574-3282) if they will be occupying laboratory or office facilities during off-shift hours. This notification will help support the emergency response personnel in the event of an off-normal event. The performance of new (i.e. first time) activities is not permitted during off-shift hours. These controls and the use of training and on-the-job experience mitigate the hazards associated with this scenario.

4.2.3 Radioactive Materials

The possibility exists that the samples that are being analyzed will be radioactive. (See MCL-7710 for guidance on sample preparation.) Handling radioactive materials could lead to personnel exposure and/or contamination of equipment/property. This hazard is controlled by engineering, administrative and PPE controls. The engineering controls are the methods by which the samples are prepared. (All RAD samples are prepared in a radiological area and are surveyed prior to removal from the area.) The sample is firmly secured onto the sample platform. For proper analysis the sample has to be stable and firmly in place. The administrative controls are enacted by the HP organization. The HP technician must first establish a radioactive materials storage area (RMSA). This involves the survey of the instrument before and after the sample has been analyzed and the surrounding area. This HP support ensures that radiological material has not come loose during the analysis. The PPE containment involves the use of a

sample transfer box from the radiological area to the temporary RMSA at the instrument, the use of gloves while handling the sample, the skirting of the instrument area with yellow (RAD) plastic or tyvek, and radiological disposal of all PPE and lab supplies used in the RMSA.

4.2.4 Ionizing Radiation

This hazard recognizes the fact that x-rays are produced by the x-ray tube. These x-rays could be a potential source for personnel exposure. This hazard is mitigated by engineering controls. The basic requirements for the production of x-rays require the safety interlock system of the instrument to be operational. This instrument is surveyed for x-ray leakage. This means that it is not possible for a person to place their hand in, at, or near the source of x-ray production. It is also recognized that the source of the ionizing radiation can be totally removed by shutting off the x-ray gun.

4.3 Classified Work

Follow all guidance provided in the MCLinc Facility Security Plan (MCL-7706) for performing classified work.

4.4 Emergency Shutdown

The safest, most direct method of shutting the instrument off should be posted in clear plain sight on the front of the instrument. The instructions should be in large print, signed, dated, and laminated.

5.0 ENVIRONMENTAL AND WASTE MANAGEMENT CONCERNS

5.1 Waste Minimization Methods

Sample preparation — use of smallest possible beaker or test tube for cleaning samples or equipment (e.g. tweezers, spatula). Use only a portion of a paper towel or wipe as needed.

5.2 Waste Disposal Methods

All RCRA/TSCA/RAD waste generated by this process shall be disposed of in accordance with MCLinc policies (MCLinc Chemical Hygiene Plan, MCL-7702).

5.3 Environmental Risks

No appreciable environmental risks are noted at this time for XRD operation.

6.0 QUALITY AND PERFORMANCE DOCUMENTATION

6.1 Quality Assurance Documentation

The following information shall be documented in the time period stated. This information will provide direct documentation of the performance (calibration) parameters affecting the quality of the output (results) of the instrumentation. The documents resulting from these QA procedures will be kept in room *A108*.

Diffraction Calibration (monthly): A series of peaks from 20 to 90 degrees two theta will be used to measure position and linearity of the goniometer. A regression analysis will be performed on the resulting diffractogram. A more detailed discussion can be found in Appendix A.

Diffraction Resolution (monthly): A plot of the degrees two theta range from 65 to 70 will be observed for the split of the five peaks that make up this region of the quartz standard diffractogram. An example of this region is in Appendix A.

Detector Performance - (monthly): A series of low and high angle diffraction peaks will be used to track any variation in peak intensity with time. An example of this tracking is in Appendix A.

6.2 Performance Documentation

The following information shall be documented on the time period stated and after shutdown periods and maintenance. This information will document the scheduled maintenance, non-scheduled maintenance, and root cause for the instrumental non-conformance. The documents resulting from these performance procedures will be kept in room *A108*.

Scheduled instrument maintenance (per event): A copy of the paper work provided by the vendor should be kept in chronological order. Any information or work which has been provided by the vendor in response to questions or operational abnormalities that is not clearly documented in the vendor's paperwork should be documented and attached to the vendor's paperwork.

Non-scheduled instrument maintenance (per event): A copy of the paper work provided by the vendor should be kept in chronological order. Any information or system work which is not clearly documented on the vendor's paperwork or work instructions provided over the telephone should be documented.

Instrument calibration non-conformance (per event): The actions required to bring the instrument back into compliance with operating specifications as noted in section 6.1 should be documented.

6.3 Vendor Manuals

Vendor manuals form the basis of the documentation for operating information. These manuals in combination with vendor/professional training and on-the-job training should allow the principle operator to safely, properly, and fully operate the instrumentation.

Vendor manuals shall be kept in good condition and be readily available during instrument operation.

6.4 Data Tracking

Diffraction patterns collected should be stored on disk in the raw data format.
All diffraction patterns should be given a unique filename. The file name should be logged with information concerning the sample ID number, the operating conditions, the disk storage ID number, and the date.

7.0 REFERENCES

MCLinc *Chemical Hygiene Plan* (MCL-7702)

MCLinc *Quality Assurance* (MCL-7701)

MCLinc *Sample Preparation Guide* (MCL-7710)

Appendix A
Quality Assurance Documentation

Quality assurance documentation for the XRD is obtained on a monthly basis. The quartz standard from Philips Electronic Instruments should be run at 40 k*V* and *35 mA*. The program used to do the standard run covers the range 20 to 90 degrees two theta in 0.01 degree steps. Each step is counted for 2.5 seconds and the peak location is calculated by the XRD computer using the centroid second derivative technique. The plots of the diffractogram and the computer printout are archived in Room *A108*. Subsequent computer analysis of the data is done using Microsoft Excel.

There are three areas of calibration interest. The first of these is the two theta position of the diffractometer. To evaluate this parameter of XRD operation a series of peaks encompassing the general range from 20 to 90 degrees two theta are compared to the standard quartz profile. The differences between the monthly run and the standard peak locations are tabulated and and a linear regression analysis is performed on the diffractogram results (Figure 1). The consistency of the slope and intercept from the linear regression is tracked. Should the regression analysis show inconsistency, the root cause will be determined and actions will be taken to correct the inconsistency.

The second area of interest is the check of x-ray tube and detector performance. A series of low and high angle diffraction peaks is used to tract any variation in peak intensity. An example of this tracking is shown in Figure 3. If the intensity falls below an acceptable level, the root cause will be determined and actions will be taken to correct the variance from acceptable operating conditions.

The third area of interest is the check of detector performance. A plot of the 65 to 70 degrees two theta region versus intensity will be observed for the split of the five peaks that make up this region of the quartz standard diffractogram. An example of this region is shown in Figure 2. In addition, the Ka1 peak FWHM at 59.2 degrees two theta will be determined and plotted for variation. If the resolution increases to an unacceptable level, the root cause will be determined and actions will be taken to correct the variance from acceptable operating conditions.

Figure 1
Linear Regression Analysis Plot

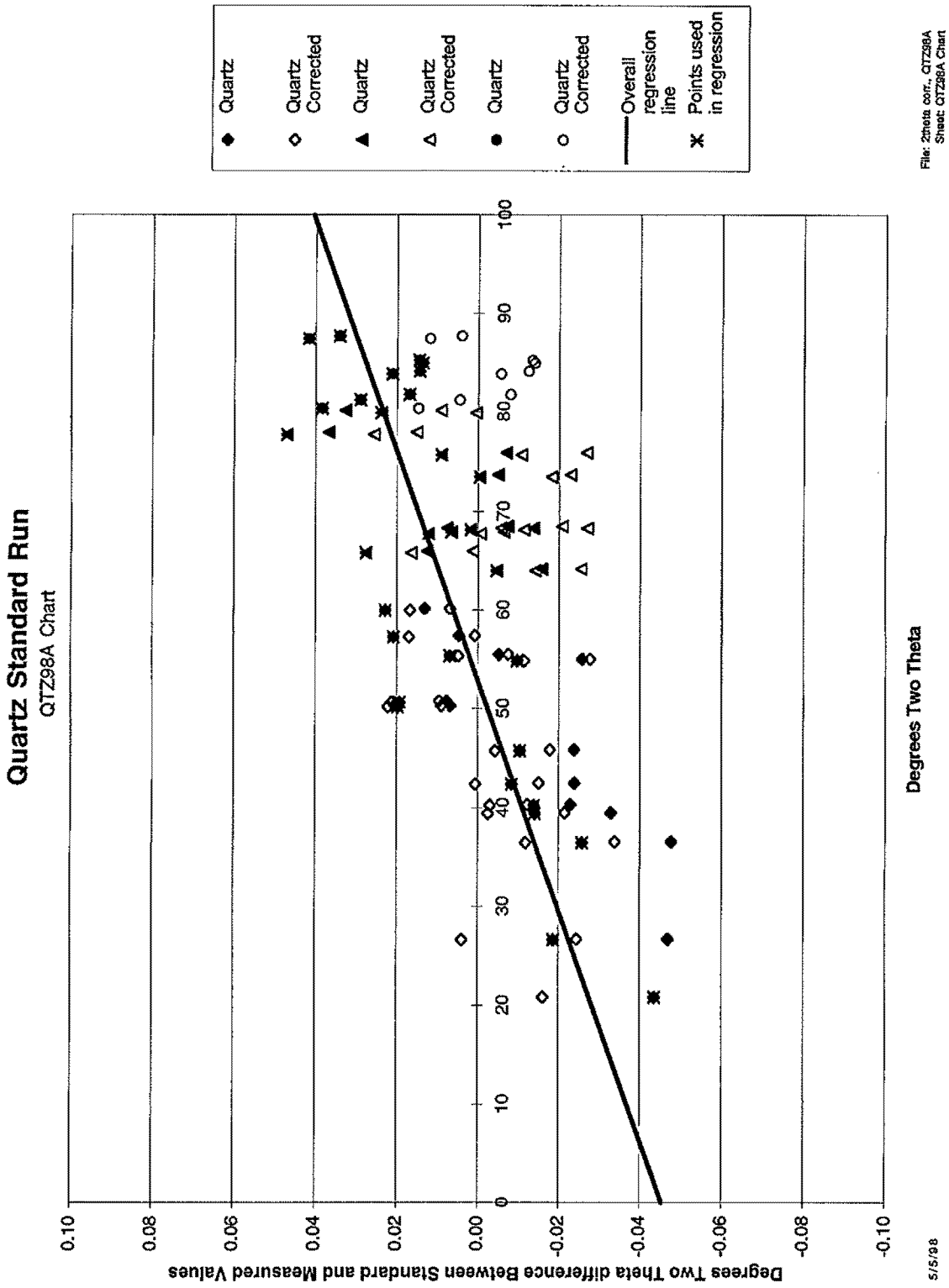


Figure 2
Example of Resolution Check

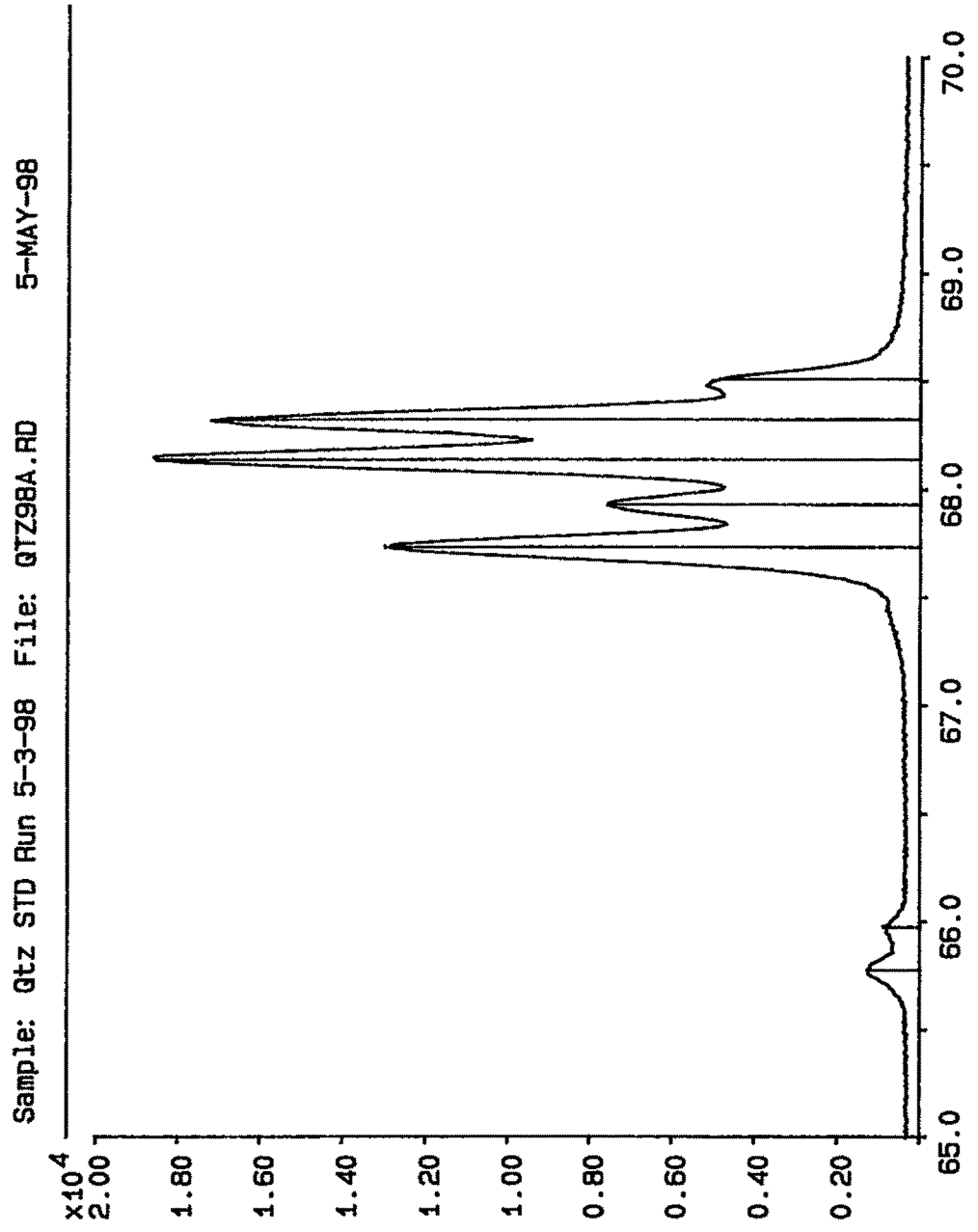
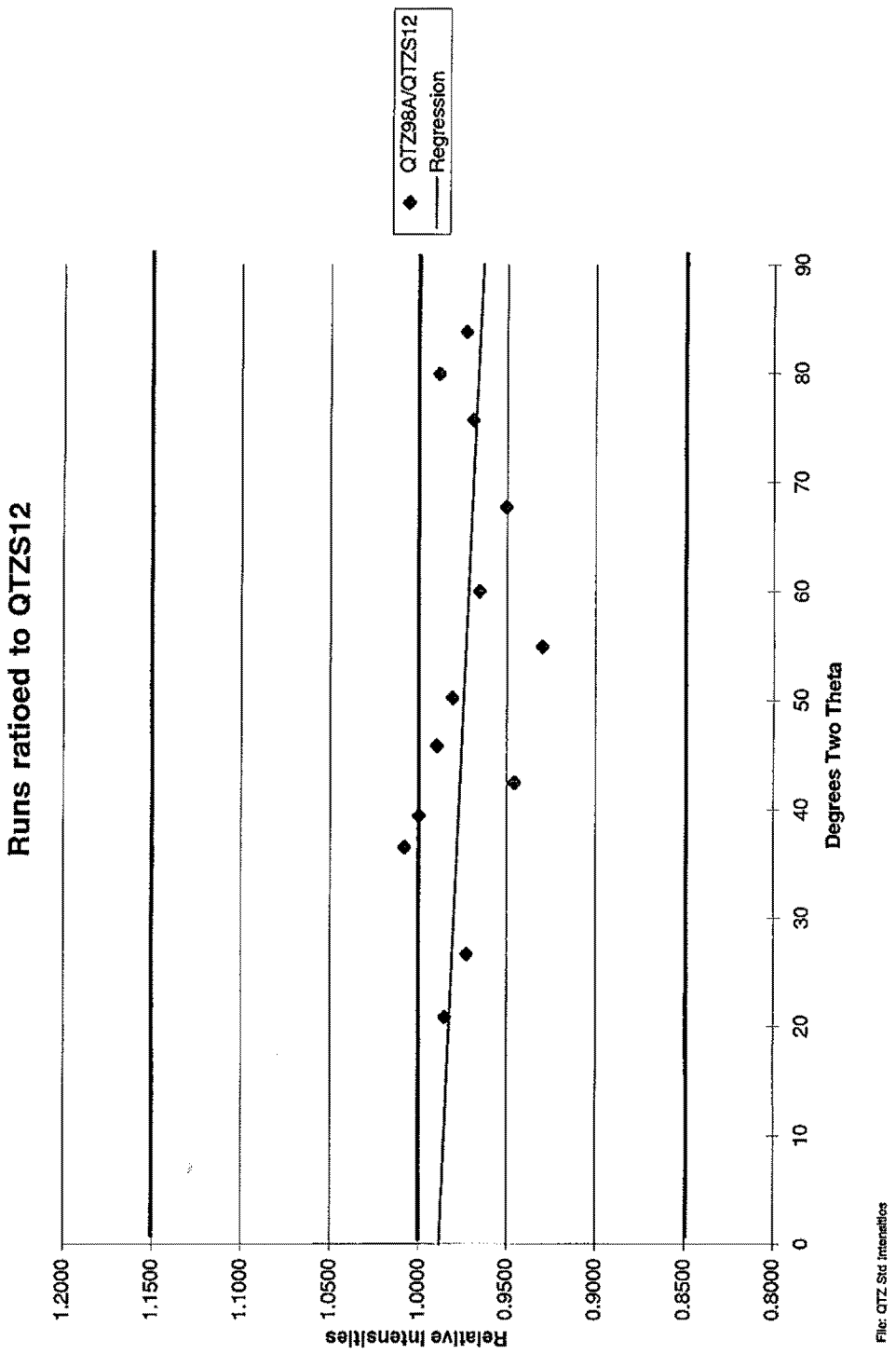


Figure 3
Example of Quality Assurance Intensity Check



MATERIALS AND CHEMISTRY LABORATORY, INC. STANDARD OPERATING PROCEDURE	
Determination of Uranium by a Modified Davies-Gray Titration: Materials and Chemistry Laboratory, Inc.	Approved: MCLinc President — Date: 9/15/11 Quality Assurance Officer — Date: 9/12/11

1.0 PURPOSE

This procedure applies to samples of uranium compounds of the nature UxFz,, UxOyFz, UxOy and others relating to uranium contaminated scrap materials where interfering elements are kept to a minimum.

2.0 SCOPE

This procedure may also be applied to determine levels of uranium in aqueous and solid samples.

3.0 ROLES AND RESPONSIBILITIES

MCLinc analyst is responsible for performing the analysis on the samples per this procedure, reviewing the results, and reporting any problems.

The Operations Manager or Project Manager represents the first level of management and provides project oversight.

4.0 MATERIALS AND APPARATUS

- Platinum wire: 12"
- Orion Ag-AgCl Half-Cell Single Junction Reference Electrode
- *Thermo Scientific Orion Star pH/ISE Benchtop Meter* or equivalent
- Micro buret-2 ml, 0.002 ml graduations, Gilmont GS-1200A
- Magnetic stirrer
- Teflon coated stir bars
- Hot plate
- Fume hood
- Muffle or tube furnace

- Assorted laboratory glassware – cleaned in laboratory detergent solution and rinsed well in DI H_20
- Platinum or quartz boats
- Thermometer

5.0 REAGENTS

- Sulfuric acid (H_2SO_4): 96%, concentrated.
- Sulfamic acid (H_2NSO_3H): reagent grade.
- Phosphoric acid (H_3PO_4): 85%, concentrated.
- Ferrous sulfate ($FeSO_4.7H_2O$)), granular or crystal.
- Nitric acid (HNO_3): 70%, concentrated.
- Ammonium molybdate [$(NH_4)_6Mo_7O_{24}.4H_2O$], crystals.
- Vanadyl sulfate ($VOSO_4.nH_2O$), 99% pure.
- Potassium dichromate ($K_2Cr_2O_7$), Primary Standard Grade.
- Triuranium octaoxide, (U_3O_8), highly pure.
- Distilled or deionized water.
- Chromic acid for glassware cleaning.
- Sodium hydroxide, (NaOH), pellets or other caustic chemical for acid neutralization
- Laboratory detergent.

6.0 REAGENT PREPARATION

A. 1 M Sulfuric acid solution:

1. To a 2-liter volumetric flask, add ~ 1000 ml DI H_2O.
2. Carefully, while holding flask under the cold water faucet, add 110 ml of concentrated H_2SO_4 while swirling.
3. Allow to cool and then dilute to volume with DI H_2O.

Shelf life: ~ 6 months.

B. 1.5 M Sulfamic acid:

1. To a 1-liter volumetric flask, add 145.5 g of sulfamic acid and ~ 800 ml of DI H_2O.
2. Stir on a magnetic stirrer with gentle heat sufficient to dissolve the solids.
3. Cool and dilute to volume with DI H_2O.

Note: 100 ml of this solution is used in the preparation of the reagent in 6.0 D.

Shelf life: ~ 6 months.

C. 1 M Ferrous sulfate solution:

1. To a 100 ml volumetric flask, add ~65 ml DI H_2O.
2. Carefully add 10 ml of concentrated H_2SO_4.
3. Add 28 g of $FeSO_4.7H_2O$.
4. Carefully shake to dissolve, cool, and dilute to volume with DI H_2O

Shelf life: 2 days

D. Nitric-sulfamic acid solution with ammonium molybdate:

1. To a 1 liter storage bottle, add 400 ml of DI H_2O.
2. Add 4.0 g of $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ and dissolve.
3. Add 500 ml of concentrated HNO_3 and mix.
4. Add 100 ml of the sulfamic acid solution previously prepared in 6.0 B and mix well.

Shelf life: ~ 6 months

E. 0.027 N Potassium dichromate standard solution:

1. Dry *NIST* SRM 136*F* $K_2Cr_2O_7$ or equivalent primary standard grade for *2* hrs at 110 °C.
2. Cool in dessicator.
3. Weigh out about 1.325 g (accurately to 4 decimal places) of the dichromate for 1 liter of solution corrected for the assay.
4. Add the weighed dichromate to a 1-liter volumetric flask (calibrated), dissolve and dilute to volume with DI H_2O.
5. Transfer the solution to a 1 liter glass bottle.
6. Calculate the normality of the solution.

Normality ($K_2Cr_2O_7$) = mass(g) x assay x 1 mol/294.1844 g x 6 eq/mol x 1/vol. flask (L)

Shelf life: indefinite.

F. 0.008 N Potassium dichromate solution:

1. Dissolve 0.39 g $K_2Cr_2O_7$ in a 1-liter volumetric flask with DI H_20.
2. Bring to volume with DI H_2O.
3. This solution is used to oxidize impurities in the phosphoric acid, its normality does not have to be precise.

Shelf life: indefinite

G. Uranium standard solution, ~ 340 ppm U in 10% HNO_3.

1. Place a small quantity of NBS 950b U_3O_8 in a quartz or platinum boat.
2. Insert the boat into the tube furnace at 800 deg C for 1 h.
3. Cool in a dessicator to room temperature.
4. Weigh out ~0.4 g of U_3O_8 in a 200 ml tall form beaker.
5. Add 50 ml 10% HNO_3 and 50 ml conc. HNO_3.
6. Heat gently on a hot plate to dissolve.
7. Cool and transfer to a 1 liter calibrated volumetric flask using DI H_2O.
8. Add 45 ml of conc. HNO_3.
9. Dilute to volume with DI H_2O.
10. Transfer the solution to a 1 liter glass bottle.

Shelf life: indefinite

Calculation of uranium concentration in the standard solution.:

0.4000 g U_3O_8/1 L x 0.99968 g U_3O_8/1.00000 g U_3O_8 x 0.848001 g U/ 1.000000 g U_3O_8 x 1000 mg U/1 g U = 339.09 mg U/L

Standards are reanalyzed when the deviation from the accepted value exceeds 0.1 mg.

H. Uranium standard solution, ~ 42 ppm U in concentrated H_3PO_4.

1. Place a small quantity of NBS 950b U_3O_8 in a quartz or platinum boat.
2. Insert the boat into the tube furnace at 800 deg C for 1 h.
3. Cool in a dessicator to room temperature.
4. Weigh out ~0.05 g of U_3O_8 in a 200 ml tall form beaker.
5. Add ~25 ml concentrated H_3PO_4.
6. Heat gently on a hot plate to dissolve.
7. Cool and transfer to a 100 ml volumetric flask using concentrated H_3PO_4.
8. Dilute to volume with concentrated H_3PO_4.

Shelf life: U+4: Analyze within a week, total U indefinite

Calculation of uranium concentration in the standard solution.:

0.0500 g U_3O_8 x 0.99968 g U_3O_8/1.00000 g U_3O_8 x 0.848001 g U/ 1.000000 g U_3O_8 x 1000 mg U/1 g U = 42.386 mg U/L

Standards are reanalyzed when the deviation from the accepted value exceeds 0.1 mg.

7.0 PROCEDURE

A. Total U for samples dissolved in dilute HNO_3

1. To 300 ml tall form beaker add the following in order:
   a) Magnetic stir bar.
   b) 15 ml sample (pipetted).
   c) 3 ml conc. H_2SO_4 and swirl.
   d) 5 ml 1.5 M sulfamic acid and swirl.
   e) 40 ml conc. H_3PO_4 down the beaker walls and swirl.
   f) 3 ml DI H_2O and swirl.
   g) 1 ml 0.008 N $K_2Cr_2O_7$ and swirl.
   h) 5 ml 1 M $FeSO_4$ and swirl. (Allow 30-60s reaction time, adjust temperature to 40-43 deg C during this time period).
   i) 10 ml nitric-sulfamic acid solution and swirl. (Allow 3 min reaction time, weigh out vanadyl sulfate and prepare electrodes during this time period).
   j) 100 ml 1 M H_2SO_4 (wash down thermometer).
   k) 100 mg – 120 mg vanadyl sulfate.

2. Insert the electrodes and immediately titrate with 0.027 N $K_2Cr_2O_7$. (The endpoint is between 590-620 mv.) Rapidly add titrant until ~520 mv is reached. Then add titrant in 0.01 ml or 0.002 ml increments depending on uranium concentration, and record the potential at each addition of titrant. Use the second derivative method of calculating the endpoint.)

3. Place the remaining solution in the appropriate waste container.

B. Total U for samples dissolved in concentrated H_3PO_4.

1. To 300 ml tall form beaker add the following in order:
   a) Magnetic stir bar.
   b) 15 ml sample (pipetted).
   c) 3 ml conc. H_2SO_4 and swirl.
   d) 5 ml 1.5 M sulfamic acid and swirl.
   e) 28 ml conc. H_3PO_4 down the beaker walls and swirl.
   f) 11 ml DI H_2O and swirl.
   g) 1 ml 0.008 N $K_2Cr_2O_7$ and swirl.
   h) 5 ml 1 M $FeSO_4$ and swirl. (Allow 30-60s reaction time, adjust temperature to 40-43 deg C during this time period).
   i) 10 ml nitric-sulfamic acid solution and swirl. (Allow 3 min reaction time, weigh out vanadyl sulfate and prepare electrodes during this time period).
   j) 100 ml 1 M H_2SO_4 (wash down thermometer).
   k) 100 mg – 120 mg vanadyl sulfate.

2. Insert the electrodes and immediately titrate with 0.027 N $K_2Cr_2O_7$. (The endpoint is between 590-620 mv.) Rapidly add titrant until ~520 mv is reached. Then add titrant in 0.01 ml or 0.002 ml increments depending on uranium concentration, and record the potential at each addition of titrant. Use the second derivative method of calculating the endpoint.)

3. Place the remaining solution in the appropriate waste container.

C. Procedure for U^{+4} (sample must be dissolved in concentrated H_3PO_4)

1. To 300 ml tall form beaker add the following in order:
   a) Magnetic stir bar.
   b) 15 ml sample (pipetted).

Allow the beaker to stand while the rest of the reagents are added to a separate clean beaker.

2. To a separate clean 250 ml beaker, add:
   a) 15 ml conc. H_3PO_4 and swirl.
   b) 3 ml conc. H_2SO_4 and swirl.
   c) 5 ml 1.5 M sulfamic acid and swirl.
   d) 13 ml conc. H_3PO_4 down the beaker walls and swirl.
   e) 11 ml DI H_2O and swirl.
   f) 1 ml 0.008 N $K_2Cr_2O_7$ and swirl.
   g) 5 ml 1 M $FeSO_4$ and swirl. (Allow 30-60s reaction time, adjust temperature to 40-43 deg C during this time period).
   h) 10 ml nitric-sulfamic acid solution and swirl. (Allow 3 min reaction time, weigh out vanadyl sulfate and prepare electrodes during this time period).
   i) 100 ml 1 M H_2SO_4 (wash down thermometer)..
   j) Add this solution to the beaker containing the pipetted sample (steps 1a-1b).
   k) Add 100 mg – 120 mg vanadyl sulfate.

3. Insert the electrodes and immediately titrate with 0.027 N $K_2Cr_2O_7$. (The endpoint is between 590-620 mv.) Rapidly add titrant until ~520 mv is reached. Then add titrant in 0.01 ml or 0.002 ml increments depending on sample size, and record the potential at each addition of titrant. Use the second derivative method of calculating the endpoint.)

4. Place the remaining solution in the appropriate waste container.

8.0 CALCULATIONS

Sample Titration Data showing 2nd derivative method:

A Volume (ml)	B Potential (mV)	C d(B)/ d(A)	D $d^2(B)/ d^2(A)$
0.000	559		
0.008	576		
0.010	583		
		\	
		7,500	
		/ \	
0.012	598		+ 4,500
		\ /	
		12,000	
		/ \	
0.014	622		- 2,500
		\ /	
		9,500	
		/	
0.016	641		

Endpoint = 0.012 ml + 0.002 ml [4,500 / (4,500 + 2,500)] = 0.01329 ml

Sample Calculation for the Amount of Uranium:

[0.01329 ml - 0.0024 ml (blank)] x 0.027039 meq/ml x 1 mmol/ 2 meq x 238.03 mg U/ 1 mmol U = 0.035 mg U

Reporting limit – The reporting limit for this procedure is the amount of uranium corresponding to 0.005 ml of titrant after blank correction.

9.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Because all materials utilized in this procedure are potentially radioactive sources, all samples, waste, and standards will be appropriately labeled and handled according to MCL-7718 and MCL-7715.

The waste will be minimized by using small volumes and minimizing quantities utilized for sample preparation and standards preparation. Materials for disposal will be segregated and properly labeled. Where possible, the waste will be reduced by known treatment methodologies.

Rad waste will be measured and documented and where necessary turned over to an approved commercial handling and disposal service.

10.0 REFERENCES

1. W. Davies and W. Gray, "A Rapid and Specific Titrimetric Method for the Precise Determination of Uranium Using Iron (II) Sulfate as Reductant,"Talanta" 11, (1964), p. 1203.

2. Eberle et al., "Titrimetric Determination of Uranium in Product, Fuel, and Scrap Materials After Ferrous Ion Reduction in Phosphoric Acid," New Brunswick Laboratory Progress Report No. 252, July, 1970.

3. R.J. Jarabek, Transport Measurements of UF_5 Using a Precision Analysis for U^{+4}, K/PS-5017, Martin Marietta Energy Systems, Inc., Oak Ridge Gaseous Diffusion Plant, April 2, 1984.

4. D.A. Skoog and D.M. West, Fundamentals of Analytical Chemistry, Holt, Reinhart, and Winston, Inc., pp. 550-554, 2nd ed., 1969.

EBERLINE SERVICES, INC.
STANDARD OPERATING PROCEDURES

Eberline Analytical
Oak Ridge Laboratory
Analytical Procedure

AP-001

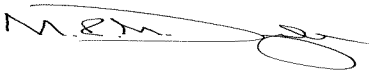
Sample Preparation (I): Pre-Chemistry

AUTHORIZATION AND APPROVAL STATEMENT

This **Eberline Analytical** - Oak Ridge Laboratory, Analytical Procedure,
Sample Preparation (I): Pre-Chemistry
is authorized and approved in its entirety by:

Saba Arnold Seaver
Quality Assurance Manager

Date: October 23, 2014



Michael R. McDougall
Laboratory Manager

Date: October 23, 2014

1.0 PURPOSE, SCOPE, AND APPLICABLE MATRICES

1.1 The purpose of this procedure is to provide direction for physical preparation of environmental samples prior to any chemical digestion or analysis.

1.2 The scope of this procedure is limited to the initial drying, grinding and physical preparation of solid or liquid samples as may be necessary.

1.3 This procedure is applicable to many solid and liquid matrices. There may be some sample matrices that will require deviations from this procedure. Individual deviation forms will cover these deviations.

1.4 Because of the volatility of some radioisotopes, it may be necessary to remove aliquots from some samples prior to physical processing. In the case of solid samples requiring determination of Iodine-131/129, Technetium-99 or Tritium, the analyst should refer to the aliquot sheet or consult with the Technical Director or Laboratory Manager for instructions for removal of a sample aliquot prior to processing.

2.0 DETECTION LIMITS

Not applicable. Refer to the specific counting or analytical methods.

3.0 SUMMARY OF TEST METHOD

3.1 A significant portion of the samples received at the Eberline Analytical Oak Ridge laboratory require physical preparation prior to analysis either to assure the homogeneity of the samples or to make them more amenable to other required chemical or counting procedures. After removal of aliquots for analyses of volatile analytes, samples are dried in a drying oven and/or ashed in a muffle furnace. After drying or ashing, samples are pulverized using a rotary plate mill and/or jaw crusher depending on the size of the individual pieces of material or the hardness of the dried sample.

3.2 Soil or solid sample preparations may differ from the referenced method in the following ways:

- Soil or liquid samples that are not dried and are analyzed as is or as received.
- Solid samples preparation shall be denoted within the laboratory technician's notes as, analyzed as received, WET or analyzed from dried, pulverized and homogenized mass.
- Solid samples that cannot be pulverized or homogenized. These types of samples include metal or refractory materials, samples that are reactive to heat caused by drying and or pulverization and samples that contain volatile radionuclides such as Tritium.
- Soil samples that contain high levels of organic material such as asphalt, tar, etc.
- Soil samples which are counted by gamma spectroscopy in special geometries.
- Water samples that are not filtered prior to analysis unless specifically directed by the client
- There are no specific deviations from references in Section 18 other that warrant addressing in this section.

4.0 DEFINITIONS

4.1 MSDS: Material Safety Data Sheets

4.2 HOC: High Organic Content

4.3 HMC: High Moisture Content

4.4 TDS: Total Dissolved Solids

4.5 TSS: Total Suspended Solids

4.6 TOC: Total Organic Carbon

5.0 INTERFERENCES

Matrices may not be amenable to all preparation techniques, e.g., attempting to crush or grind samples containing tar or large metal pieces. Consult the Technician Supervisor or Technical Director if there is any doubt as to the utility of this procedure.

6.0 SAFETY

Laboratory chemical and general safety shall be conducted as required within *EberlineAnalytical Oak Ridge Laboratory, Chemical Hygiene/Health and Safety Plan ,Latest Version*

Laboratory radiation safety shall be conducted as required within *Eberline Analytical Radiation Protection Plan and Attachments ,Latest Version*

Waste management and sample return shall be conducted as required within *Eberline Analytical Waste Management Plan ,Latest Version*

6.1 Housekeeping

6.1.1 All work areas shall be kept as clean as possible at all times and the entire work area shall be cleaned at the conclusion of the last shift of the day.

6.1.2 Minimize unnecessary items and clutter in the work space.

6.1.3 Promptly clean any spills that occur using the guidance contained in the Emergency Action Plan, Spill Response Procedure and support of the Radiation Safety Officer and Health and Safety Officer if necessary.

6.2 Clearly label all sample containers (beakers, bottles, c-tubes etc.) with the work order number, analysis fraction, and analyte identification information such as Total Sr , Iso-U , or some other recognizable wording.

6.3 Any labels that identify the hazards associated witha particular sample container at the time of receipt will remain affixed to that container AND to ALL subsequent sub sampling from, and disposal of, that container.

6.4 Dispose of all waste in the appropriate containers as directed by the Waste Management Pla n.

6.5 Dispose non -rad waste in appropriate containers, DO NOT PUT NON -RAD WASTE INTO RAD WASTE CONTAINERS.

6.6 Personal protective equipment for this procedure shall consist of a lab coat or protective apron, safety glasses or goggles and chemical resistant labor atory gloves.

6.7 No loose or baggy clothing shall be worn on the upper portion of the technicians body when operating grinding and crushing equipment. The technician s lab coat should be buttoned and rubber bands, tape, or some other method should be used to restrict the movement of the lab coat sleeves so that they will not become entangled in moving machinery.

7.0 EQUIPMENT AND SUPPLIES

7.1 Balance (0 to 3600 grams capacity)

7.2 Drying oven

7.3 Mortar and pestle

7.4 Muffle furnace

7.5 Bico Pulverizer

7.6 Sieves

7.7 Wood Chipper

7.8 Assorted laboratory glassware

7.9 Assorted stainless steel or aluminum pans

7.10 Assorted Petri dishes

7.11 Bico Jaw Crusher (Chipmunk)

7.12 Clean, dry grinding sand

7.13 The laboratory may use pre-cleaned disposable plastic lab ware as appropriate and applicable to this or any other analytical procedure. Disposable plastic ware will be disposed of in the appropriate waste container after use.

8.0 REAGENTS AND STANDARDS

There are no reagents or standards used in this procedure.

9.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

9.1 Sample collection and preservation is not the responsibility of the laboratory and is not applicable to this procedure. Upon receipt of water samples, the laboratory may preserve/pH adjust the samples depending on the composition of the sample and the requested analysis.

9.2 Unless otherwise directed by the client, after receipt, all soil, solid, water, and vegetation samples will be segregated according to preliminary activity scans and stored in a secure, climate controlled location. Tissue samples will be stored in a freezer prior to analysis.

10.0 QUALITY CONTROL

10.1 One Laboratory Control Sample (LCS) shall be analyzed with every 20 samples. The LCS will be prepared and analyzed the same way and along with the analysis batch for the same analytical parameter.

10.2 One analysis blank shall be analyzed with every 20 samples. If there are less than 20 samples per analysis batch, then one blank per batch shall be analyzed.

10.3 A minimum of one or a designated number of client samples shall be duplicated with every 20 samples (one sample for eve ry 10 client samples will be duplicated for RCRA or SW846 analyses). If there are less than 20 samples per analysis batch, then a minimum of one or a sufficient number of duplicates to meet client criteria shall be analyzed per analytical batch. Where the matrix type, limited sample volume or other special considerations preclude this as a viable option, a replicate analysis will be used for QC evaluation.

10.4 A matrix spike composed of a sample spiked with a standard containing at least one of the isotopes in question (NIST traceable or equivalent) shall be run with each sample batch analyzed for Tritium or Gross Alpha/beta as required.

10.5 Other client specific requirements may supersede these requirements

11.0 CALIBRATION AND STANDARDIZATION

11.1 Oven Temperature Calibrati on Check

11.1.1 Oven shall be visually inspected weekly to ensure that the hinges and latches are not compromised. Document the weekly inspection via the Weekly Oven/Furnace Inspection & Verification form posted by the oven.

11.1.2 Check oven temperatures weekly using hand held laser sensing temperature devices. If the temperature is not in the general area of the setting, notify the laboratory manager for corrective measures. Document the weekly temperature check via the Weekly Oven/Furnace Inspection & Verification form posted by the oven.

11.2 Grinder Plate Setting

11.2.1 While it is not possible to size each sample, the grinder setting can be adjusted prior to grinding a set of samples by using the cleaning sand as follows:

- Obtain an appropriately sized sieve, either 40 or 100 mesh
- Grind approximately 25 grams of sand and check the size using the appropriate sieve.
- If the gap between the grinder plates needs to be adjusted, loosen the adjustment screw by turning the large, handled retaining nut.
- Then turn the small, round k nob on the end of the adjustment shaft either clockwise or counter-clockwise to decrease or increase the sample grinding size as needed.
- Once the cleaning sand is the appropriate size, retighten the retaining nut , clean the pulverizer and proceed to grind the samples.

11.3 There are no standardized carriers used in this procedure.

11.3.1 The calibration check for balances is covered in procedure MP -010.

11.3.2 The use, maintenance, and volume verification of the mechanical pipettes is covered in MP - 025

12.0 PROCEDURE

12.1 Soil Samples:

12.1.1 This portion of the procedure is designed for the initial processing of environmental level soil samples and to serve as a reference for other solid matrices in this procedure. The analyst should check with the Technical Director or Laboratory Manager bef ore using the Bico Pulverizer or Chipmunk rock crusher to process the samples in any work order contained in a red folder (potential high activity level).

12.1.2 Check all sample identification numbers against work order documentation in order to verify that th e paperwork is accurate and complete. Immediately notify the laboratory Project Manager in the event an error is noted.

12.1.3 Check the analyses requested and internal chain of custody to see if any of the analytes are volatile (Tritium, Technetium and Iodine) or if the client has requested that a gamma spectral analysis be performed on an as received or dry basis.

12.1.4 If the volatile analytes are required, transfer the entire sample to a clean surface such as a steel bowl or a clean plastic tray under a hood.

12.1.5 Thoroughly mix the sample and then remove approximately 50 grams of material and place into a pre-labeled, appropriate container, (see 12.1.6). Also denote the analysis requirements on the outside of this container, i.e., ^{3}H, ^{99}Tc or ^{129}I as appropriate.

12.1.6 Acquire the sample for these analyses, if possible, by mixing, and cone and quartering techniques. Cone and quartering , consists of placing the entire sample on a clean surface, blending as best possible and splitting the sample in halves and quarters usin g a spatula or other straight edge apparatus until the sample is sectioned into an appropriate size; therefore, obtaining a representative fraction of the entire sample. In the event that the sample is moist and not suitable for separation of an aliquot by cone and quartering , then the technician will make the best possible effort to obtain a representative aliquot from the sample and transfer this to a suitable container.

12.1.7 In the event samples cannot be prepared using any of our standard methods, either d ue to elevated activity or other hazardous constituents, samples shall be aliquoted and analyzed with direction from the laboratory manager or technical director.

12.1.8 If a gamma analysis is requested on an as received basis, no documentation of moisture content is required.

12.1.9 Select the Aliquot/Dilution option from the LIMS menu. Transfer an appropriate aliquot to a tarred gamma counting jar. It is important to use an aliquot that is as close to a standard counting geometry as possible.

12.1.10 Depending on the size of the sample available for the gamma count, fill as appropriate based on directions from the laboratory manager, count room supervisor or technical director.

12.1.11 Weigh the filled container and, using the balance software, record this information in the LIMS aliquot spreadsheet. Seal the container using black vinyl (electrical) tape and place the sample in a plastic bag.

12.1.12 Transfer the samples with the appropriate paperwork to the counting room.

12.1.13 For soil samples that require drying, label and tare an appropriate size aluminum-drying pan, place on a balance and enter the tare weight(s) via the balance bridge into the moisture sheet, Pan Wt. of the LIMS datasheet. Add all sample mass to to tarred drying pan(s) as required to dry the entire sample. Reweigh the pan using the balance bridge function to enter Wet Wt. weights into the LIMS. If there is less than 500 grams of sample material, transfer the entire sample to the drying pan. Do not zero tare the drying pan by the balance, as the LIMS automatically corrects for tare weights. See illustration 1 below: If significant mass is required to be dried, use multiple pans.

12.1.14 Sample(s) are now ready to be dried. Place all samples into a preheated drying oven and dry at approximately 104° Celsius, (C) for 12 hours or until dry. After drying, remove samples and reweigh. Record the dry weights and document Dry Wt. , using the LIMS balance bridge as per illustration 2 below:

Illustration 1

Print Report - Send To Database

Enter Comments | Lab Deadline: 8/30/2001 | Date Received in Prep: 8/23/2001

01-08121

TRetec		Tare (g)	Gross (g)	
Fraction	Client ID	Pan Wt	Wet Wt.	Dry Wt.
04	RN9000804S082006	14.0670	192.9820	
05	RN9000804S082007	14.0590	191.9620	
06	RN9000804S082008	14.0130	533.8030	
07	RN9000804S082009	14.0500	133.8510	
08	RN9000803S081601	14.1240	288.4320	
09	RN9000803S081602	14.0000	1123.3680	
10	RN9000803S081603	14.0140	571.1280	
11	RN9000803S081604	14.0740	449.7690	
12	RN9000803S081605	14.0350	395.7730	

12.1.15 For soil/solid samples requirin g accelerated turn -around times and which also need drying, preheat the muffle furnace to 200 degrees centigrade (see section 12.2). After removing any aliquots that are needed for volatile isotopes as described in 12.1.2, weigh an appropriately sized aluminum-drying pan and transfer the sample into the pan. If there is a greater sample volume than will fit in a single drying pan, remove a representative aliquot as described in 12.1.2 if the sample has not already been blended. If there is any excess sample material,

return it to the original container. Record the weight of the pan and the sample on the moisture data spreadsheet (illustration 2).

12.1.16 Place the pan in the muffle furnace for at least two hours or until dry. If the sample has significant amounts of water in it, it may be necessary to remove the sample from the oven every half-hour or so and break up the clumps of material to aid in the drying process. After the sample has dried, proceed to step 12.1.10

Note

The client should be made fully aware that the process of accelerated drying could potentially drive off some elements such as Lead and Cesium that are not normally considered to be volatile. The results for these isotopes will most likely be biased low.

12.1.17 Pulverize each sample to the appropriate mesh size using the Bico Pulverizer or the Bico Chipmunk. If the sample contains large rocks that will not fit through the inlet spout for the pulverizer, crush the samples using the Bico Chipmunk. All sample mass shall be pulverized unless other instructions are given from the laboratory manager of technical director.

12.1.18 The Bico Chipmunk is currently located in the large protective plastic box. Redirect the flow of the hood system to the rock crusher by blocking the stack opening above the grinder

12.1.19 Open the plastic box. Remove the sample tray from the bottom of the rock crusher and ensure that there is no material left in the crusher.

12.1.20 If there is no residual material, return the sample tray to the crusher and switch the machine on using the motor starter located on the wall behind the plastic box.

12.1.21 Slowly feed the sample into the crusher. There is always a possibility that pieces will be thrown out of the crusher. Watch for this so that they can be returned to the crusher.

12.1.22 As soon as the last of the sample has been poured into the crusher allow sufficient time for complete crushing of the sample, audible crushing sound will cease at completion.

12.1.23 The material in the sample tray should be small enough to pass through the grinders sample port.

12.1.24 If the sample contains organic or flammable materials that would require ashing, see the instructions in step 12.2. The sample(s) may be pulverized to ~40 mesh if only gamma spectroscopy is required. The sample(s) shall be pulverized to ~100 mesh for all other analytical chemistry parameters either alone or with gamma analysis. Unless instructed otherwise by the client, it is laboratory policy to pulverize all the dried sample material.

12.1.25 To use the Bico Pulverizer, loosen and flip back the latches on either side of the lefthand end of the pulverizer, raise the top and open the end of the grinder to ensure that it is clean and there is no sample material in the grinder.

12.1.26 Close the grinder and start it running by pushing the on button on the motor starter nearest the grinder hood and pulling out on the red button mounted on the front of the grinder hood. Slowly pour the sample material into the spout on the left-hand end of the grinder. If there are pieces which will not fit through the grinder spout, these must be crushed using the Bico Chipmunk rock crusher (12.1.10).

12.1.27 After all of the sample has been ground, shut off the grinder by pushing in the red knob. When the grinding plate stops turning, open the top of the grinding section and proceed to brush as much of the sample as possible into the sample tray.

Illustration 2

Go To Top	Edit Last Pg #	Print Report - Send To Database			Cancel - Return to Menu		Clear Data From Form	
Enter Comments		Lab Deadline	Date Received in Prep		Date Sealed		Date Returned	
01-08121		8/30/2001	8/23/2001		8/24/2001		8/25/2001	
					Special Info Codes > H: Hot, O: Organic Hazard, P: PCB Ha			
TRetec	Lionville Laboratory, Inc.	Tare (g)	Gross (g)		Net (g)		Percent	
Fraction	Client ID	Pan Wt	Wet Wt.	Dry Wt.	Wet Wt.	Dry Wt.	Liquid	Solid
04	RN9000804S082006	14.0670	192.9820	181.9580	178.9150	167.8910	6.16%	93.84%
05	RN9000804S082007	14.0590	191.9620	182.5210	177.9030	168.4620	5.31%	94.69%
06	RN9000804S082008	14.0130	533.8030	516.2230	519.7900	502.2100	3.38%	96.62%
07	RN9000804S082009	14.0500	133.8510	119.0470	119.8010	104.9970	12.36%	87.64%
08	RN9000803S081601	14.1240	288.4320	259.5170	274.3080	245.3930	10.54%	89.46%
09	RN9000803S081602	14.0000	1123.3680	1102.0430	1109.3680	1088.0430	1.92%	98.08%
10	RN9000803S081603	14.0140	571.1280	506.6030	557.1140	492.5890	11.58%	88.42%
11	RN9000803S081604	14.0740	449.7690	424.6660	435.6950	410.5920	5.76%	94.24%
12	RN9000803S081605	14.0350	395.7730	350.8130	381.7380	336.7780	11.78%	88.22%

Note

The LIMS automatically calculates net weights, percent liquids and percent solids

12.1.28 Pull the sample tray out of the grinder to remove the sample for further processing After pulverization, open the Bico pulverizer and brush all interior areas into the catch pan within the pulverizer.

12.1.29 After pulverization and removing the sample catch pan from the Bico Pulverizer, vacuum out the grinding chamber using the Rough Prep vacuum.

12.1.30 Close the grinding chamber and gr ind approximately 25 grams of clean sand, vacuum and repeat the process as necessary to assure that cleaning has been thorough.

12.1.31 Place the used sand in the waste container within the grinding hood and vacuum out the grinding chamber. If there is any sample material remaining within the grinding plates or walls of the grinding chamber, use a wire brush and the vacuum to remove the material.

12.1.32 If a mortar and pestle methods are used for grinding the sample, vacuum and then wash the mortar and pestle with a stron g soap using an abrasive scrubber. Allow both parts to dry before using again.

12.1.33 All areas shall be completely cleaned between each sample and shall be free of residual dust or any debris generated from the previous sample preparation.

12.1.34 After pulverization, blend each sample thoroughly to assure that each sample is homogeneous.

12.1.35 In the event gamma spectroscopy is required, place an appropriate amount of sample, (grams) as required for the gamma spectroscopy geometry to be counted. It is important to use an aliquot that is as close to a standard counting geometry as possible. Depending on the size of the sample available for gamma counting, See the laboratory manager, count room supervisor or technical director for appropriate geometry containers based on samp le characteristics.

Weigh the filled container and, using the balance software, record this information in the LIMS Aliquot/Dilution spreadsheet in the aliquot column under the aliquot data section of the appropriate work order spreadsheet. Seal the container using black vinyl (electrical) tape and place the sample in a plastic bag.

Illustration 3

Go To Top | Edit Last Pg # | Print Report - Send To Database | Cancel - Return to Menu | Clear Data From Form

Enter Comments	Lab Deadline	Date Received in Prep	Date Sealed	Date Returned	Technician
01-08121	8/30/2001	8/24/2001	8/25/2001	8/26/2001	TMILES

Special Info Codes > H: Hot, O: Organic Hazard, P: PCB Hazard, R: Rush, T: Other (see comments)

TRetec	Lionville Laboratory, Inc.	Tare (g)	Gross (g)		Net (g)		Percent		Gamma		Special
Fraction	Client ID	Pan Wt	Wet Wt.	Dry Wt.	Wet Wt.	Dry Wt.	Liquid	Solid	Dry Wt.	LEPS Wt.	Info
04	RN9000804S082006	14.0670	192.9820	181.9580	178.9150	167.8910	6.16%	93.84%	128.1670		
05	RN9000804S082007	14.0590	191.9620	182.5210	177.9030	168.4620	5.31%	94.69%	123.6360		
06	RN9000804S082008	14.0130	533.8030	516.2230	519.7900	502.2100	3.38%	96.62%	473.6230		
07	RN9000804S082009	14.0500	133.8510	119.0470	119.8010	104.9970	12.36%	87.64%	73.5860		
08	RN9000803S081601	14.1240	288.4320	259.5170	274.3080	245.3930	10.54%	89.46%	206.4270		
09	RN9000803S081602	14.0000	1123.3680	1102.0430	1109.3680	1088.0430	1.92%	98.08%	710.5390		
10	RN9000803S081603	14.0140	571.1280	506.6030	557.1140	492.5890	11.58%	88.42%	417.4930		
11	RN9000803S081604	14.0740	449.7690	424.6660	435.6950	410.5920	5.76%	94.24%	360.1420		
12	RN9000803S081605	14.0350	395.7730	350.8130	381.7380	336.7780	11.78%	88.22%	285.2460		

12.1.36 In the event that radiochemistry techniques are to be conducted, after section 12.1.14, place approximately 20 grams of pulverized material into an app ropriate pre-labeled container. Place the small containers in a plastic bag. If there is excess pulverized material, place this in a new, labeled container such as a large gamma jar. Return all samples and containers to their original location in the sample storage area or transfer the samples with the appropriate paperwork to the count room or to the other appropriate location with the necessary paperwork. Document the sample(s) receipt and release information as required on the internal chain of custody form.

12.2 Ashing of Soil/Solid Samples

12.2.1 In some cases, there is sufficient vegetation, tar, or other organic materials in what has been classified as a soil , solid or sediment to make it difficult, if not impossible, to employ normal soil analysis techniq ues. Frequently, the presence of these organics makes it impossible to mix the sample effectively and take a representative aliquot for chemical analysis. With the concurrence of the laboratory manager or technical director and discussions with the client (to alert them to the potential for removal of volatile isotopes) these samples may be muffled to remove the organic components.

NOTE

Always use new beakers to ash solid or tissue samples. Never operate the muffle furnace when the laboratory is unoccupi ed. Ensure that laboratory personnel are aware of and periodically monitor the operation of the muffle furnace.

12.2.2 Open the muffle data section of the LIMS (under Lab Tech Functions), select the desired work order and enter the weight of a large clean/ne w beaker in the appropriate section (see Illustration 4). Label the dish using a green heat resistant crayon.

12.2.3 After mixing the sample and taking a representative aliquot as described in 12.1.2, transfer roughly 150 grams to the tarred container. For many s amples, it may not be possible to put this much mass in the container so use only what can be fit in a single tarred dish.

12.2.4 Weigh the filled dish and record the weight in the LIMS spreadsheet.

12.2.5 Repeat this for all other samples in the work order.

12.2.6 Set the muffle furnace as follows:

- Turn on the furnace by pressing the on-off rocker switch and check to see that the preset temperature on the display is at the desired level (650 ^{o} or 200 ^{o} for accelerated drying)
- To set the furnace for accelerated drying, adjust the temperature to 200 using the up and down arrows and leave the furnace on. To set the furnace for ashing, adjust the furnace to 650 using the up and down arrows and then switch the power off.
- Place the samples in the furnace and turn the power on.
- Check to be sure that the temperature set is still at 650^{o}. If it is not, reset using the up and down arrows. For safety reasons, do not start the muffle furnace before the end of the last shift of the day, or leave the muffle furnace in the ON position overnight.
- After the samples have cooled, remove the crucibles from the oven.

12.2.7 Open the appropriate muffle data spreadsheet and enter the post muffle weight for each sample. The LIMS will calculate the ratio of pre to post-ashing weights and use this information to calculate the pre-ashing aliquots based on a give weight of ash.

12.2.8 Examine the ashed sample to determine if it needs to be pulverized or if other physical treatment is necessary prior to preparing individual sample aliquots.

12.3 Minimal Size Samples

12.3.1 In the case where the client provides a relatively small amount of soil or solids for analysis, fifty grams for example, the above steps for soil analysis should be used as a guide with the appropriate reduction in aliquot sizes. Additionally, it may be more de sirable to use a mortar and pestle for grinding of the sample to minimize loss. The proper technique for use of the mortar and pestle is:

- Transfer the dried sample material to the mortar.
- Start grinding by firmly pressing down with the pestle and turning it. Do not pound.
- As the particles become broken down, change to a circular, rotating movement of the pestle around the mortar. See the Technical Director is necessary.

12.3.2 Most often the petri dish geometry will be used for gamma counting of small samples.

Note

In the event that multiple analyses (gamma, volatile isotopes, chemistry, etc) are requested by the client, it may be necessary to perform the analyses in a specific order to maximize the sample aliquot available for any single analysis. Consult with the Laboratory Manager or Technical Director in any case where the solid sample aliquot is significantly less than that normally seen.

12.4 Elevated Activity Samples

12.4.1 Any work order in a red folder requires instruction from the Technical Director or Laboratory Manager.

12.4.2 If it is determined that processing of the sample poses a possibility of contamination for the grinder, a sub-sample of appropriate size should be collected from the bulk sample using the

techniques in steps 12.1.2 - 12.1.5 and then ground using a mor tar and pestle according to step 12.3.

12.4.3 Prepare the appropriate aliquots as determined by the activity level of the samples. Open the aliquot/dilution section of the LIMS and enter the results in the aliquot column under the aliquot data section of the appropriate work order spreadsheet.

12.4.4 Transfer the samples with the associated paperwork either to the count room or sample storage depending on the analysis.

12.5 Vegetation Samples

12.5.1 When receiving vegetation samples, check sample numbers against the sample paperwork as detailed in 12.1.1.

12.5.2 If the client requires the analysis of volatile isotopes or gamma analysis as received , use steps 12.1.2 through 12.1.5 as a guide for the preparation of these samples.

12.5.3 If necessary due to the size of the individual pieces of vegetation shred the vegetation using scissors, a knife and cutting board or other method prior to selection of an aliquot for preparing a as received gamma sample.

12.5.4 If the analyses do not require the determination of volatile isotopes, transfer the sa mple to a tarred clean/ new beaker or ceramic dish and ash the samples according to section 12.2.

12.5.5 Transfer the appropriate ashed aliquots as detailed above.

12.5.6 Print the analysis sheet using Print Analysis Sheet function within the LIMS.

12.5.7 Collate all LIMS in formation and place into the sub -file. Place samples on a lab cart, and submit for counting by gamma spectroscopy, chemical analysis, or return to the sample storage area as appropriate.

12.6 Air filters, Gamma Isotopic

This section of the procedure is not perf ormed in the physical preparation lab.

12.6.1 Air filter samples are counted in the appropriate air filter geometry (i.e. single filter or composite filters). In the event the filter is to be analyzed on the basis of mass, weigh filter as depicted in illustrations 1, 2 and 3 above.

12.6.2 Place the air filter(s) into the appropriate counting geometry. Enter the appropriate units of measure in grams, milliliters, filter(s), or cubic feet or as required within the aliquot sheet of the LIMS. If aliquot size is in question, contact the laboratory Project Manager.

12.6.3 Print the analysis sheet using Print Analysis Sheet function within the LIMS

12.6.4 Collate all LIMS information and place into the sub -file. Place samples on a lab cart, and submit for counting by gamma spectroscopy. Rel inquish samples via the internal chain of custody form to the count-room personnel or sample storage as required.

12.7 Core Samples (Soils)

12.7.1 Requirements for the preparation of core samples may vary so these instructions should only be considered as general guidance. Consult with the laboratory Project Manager for specific instructions on how to proceed with sample preparation.

12.7.2 In the event that depth profiling (analysis of slices of the core) is not required, remove the entire sample from the sample tube (if one is present) and proceed to Section 12.1.

12.7.3 In the event that depth profiling is required for the sample, determine if the material in the sampling tube is thick enough to allow removal from the tube without deforming. If this is not the case, place the tube in one of the Laboratory freezers until the sample material is frozen solid.

12.7.4 Place a clean piece of plastic sheeting or a plastic bag, long enough to hold the entire core sample, within a dust collection hood. If the core material is relatively solid,use a wooden rod or a plastic pipe with tape over the end of the pipe to push the core sample out of the tube. If the material in the tube has been frozen, briefly heat the outside of the tube with hot water and then push the core sample out of the tube.

12.7.5 Use an appropriate cutter to slice sections off the soil core as directed by the client. Be sure to clean the cutting device between uses. Place sections in a clean prelabeled container with the corresponding sample identification. Proceed to section 12.1 .

12.7.6 Place each section or, if uncut, the entire core sample in a tarred aluminum pan. Weigh, dry, and grind the sample(s) as described in Sections 12.1.6.

12.7.7 Print the analysis sheet using Print Analysis Sheet function within the LIMS

12.7.8 Collate all LIMS information and place into the sub-file. Place samples on a lab cart, and submit for counting by gamma spectroscopy or other chemical analyses. Relinquish samples via the internal chain of custody to the count-room personnel or storage area as required.

12.8 Gamma Counting of Liquids

12.8.1 These samples are prepared in the sample preparation laboratory. All liquid samples for gamma counting are made up in polyethylene Marinelli beakers.

12.8.2 Using a clean graduated cylinder, measure the required sample volume and pour the liquid into the Marinelli beaker.

12.8.3 If the sample aliquot is larger than 500 milliliters, label a clean glass beaker of the appropriate size and transfer a carefully measured aliquot of the total sample volume to the beaker.

12.8.4 Heat the sample on a hotplate until the volume decreases to a point where more volume may be added. Continue adding additional portions of the total aliquot volume until the residual volume is slightly less than or equal to 500 milliliters.

12.8.5 Adjust the volume if necessary to bring the total volume to 500 milliliters and transfer the sample aliquot to a 500-milliliter Marinelli beaker.

12.8.6 Place the lid securely on the Marinelli beaker and seal with electrical tape. Inspect for any leakage before sending the sample for counting. Document aliquots analyzed in the LIMS spreadsheet as appropriate.

12.8.7 In the event that the available sample volume is less than 500 milliliters, measure the sample volume and transfer the sample into a Marinelli beaker. Add sufficient deionized water to reach a total volume of 500 milliliters. Record the original sample volume (prior to the addition of any deionized water) and the final volume in the LIMS system.

12.8.8 Print the analysis sheet using Print Analysis Sheet function within the LIMS

12.8.9 Collate all LIMS information and place into the sub-file. Place samples on a lab cart, and submit for counting by gamma spectroscopy. Relinquish samples via the internal chain of custody form to the count-room personnel or storage area as appropriate.

12.9 Air Filter Samples - Cellulose Filters for Isotopic Analysis, (where ashing is required).

12.9.1 Weigh and enter in the LIMS Muffle Data , section, the tare weight for a clean/new glass beaker or crucible. Place the air filter sample in a suitable sized glass beaker or crucible and place into a muffle furnace at 650° C. Using the LIMS Muffle Data section, enter weights, pre and post muffling as per illustration 4 below.

12.9.2 Heat the sample at 650° C until it is thoroughly ashed. Remove the sample from the muffle furnace, allow to cool, weigh, record post weight as above, and transfer to Sample Preparation for digestions as required.

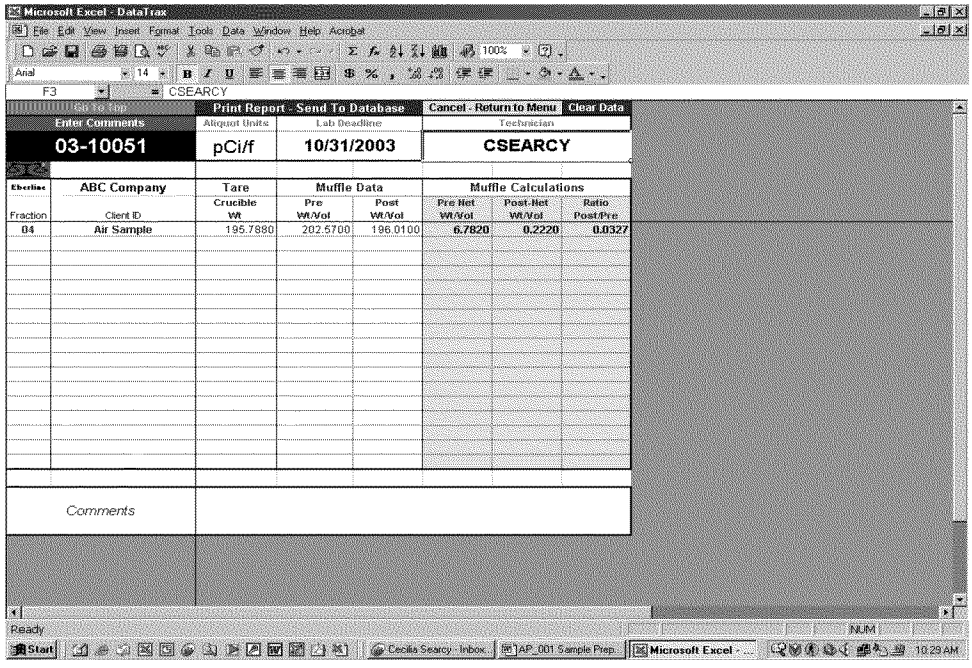
12.10 Tissue Samples

12.10.1 Cut the samples into small pieces as appropriate.

12.10.2 Weigh and enter in the LIMS Muffle Data , section, the tare weight for a clean/new glass beaker. Place the sample in a suitable sized glass beaker and place into a muffle furnace at 650° C. Using the LIMS Muffle Data section, enter weights, pre and post muffling as per illustration 4.

12.10.3 Heat the sample at 650° C until it is thoroughly ashed. Use section 11.2 as a guide for this procedure. Remove sample from muffle furnace, allow to cool, weigh, record post weight as above, and transfer sample and appropriate paperwork to the sample preparation laboratory for digestions as required.

Illustration 4



13.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

13.1 All excess sample materials, extracts, byproducts, and associated waste will be disposed of in the appropriate containers and segregated into the appropriate waste streams for final disposal according to the Waste Management Plan, WMP-01.

13.2 All laboratory activities associated with this procedure will be carried out in the fashion designed to generate the least amount of waste possible and still achieve the necessary quality of data.

13.3 The cleaning sand from the grinder will be retained in a suitable container until it can be sampled and analyzed for radiological contamination. Once the sand has been characterized for radiological and, if necessary, hazardous components, it will be added to the appropriate waste str eam.

13.4 The aluminum drying pans will be retained in a suitable container. These will be surveyed when necessary to determine if they should be added to the solid radioactive waste stream or if they can be disposed of as solid, dry waste.

13.5 Pre-cleaned disposable plastic lab ware will be placed in the appropriate waste stream following its use in the laboratory.

14.0 CALCULATIONS

14.1 The calculations in this procedure are all addition, subtraction, or ratios.

15.0 METHOD PERFORMANCE

15.1 The initial method performance shall be determined using the method detailed in procedure MP-028.

15.2 The method performance is continuously monitored using the laboratory control standards, blanks and replicates/duplicates.

16.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

It is the laboratory policy to analyze a Laboratory Control Sample (LCS), a Laboratory Method Blank (MBL), and a Duplicate (DUP) with each work order. Soil samples will be reported on a dry weight basis unless otherwise requested by the client. Work orders are unique for each client, matrix, and isotope. Specific client requirements may supersede the laboratory default criteria.

16.1 The QC measurement acceptance criterion is detailed in each counting procedure.

16.2 RCRA Methods 9310 (Gross Alpha/ Beta); 9315 (Alpha Em itting Radium Isotopes); and 9320 (Radium 228) require a sample duplicate be analyzed at a frequency of one in every ten samples.

17.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1 Sample data that is deemed unacceptable will be reanalyzed when it is not possible to relate the deficiency to a calculation or clerical error and there is sufficient sample available for reanalysis.

18.0 REFERENCES

18.1 Standard Methods, 17th Edition, 1989, Method 2540B

18.2 Gamma Emitters in Water , EPA Method 901.1 Modified, U.S. En vironmental Protection Agency, Environmental Monitoring and Support Laboratory Procedures Manual (EPA -600/4-80-032, 8/80) Cincinnati, OH.

18.3 Gamma Emitters in Soil , LANL Method ER130 Modified, Los Alamos National Laboratory Procedures Manual, Los Alamos, NM.

19.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

19.1 Validation data is available on request.

19.2 There are no tables or diagrams associated with this procedure.

20.0 DEVIATIONS FROM REFERENCED METHODS

20.1 The Lithium drifted germanium gamma detectors listed in the EP A reference procedure are now obsolete. The procedure as written uses the currently available high -purity germanium detectors.

20.2 The method as written is a combination of the referenced methods and is applicable to different matrices. For example, the EPA re ferenced method is exclusively for drinking water and while this method is applicable to drinking water, other sample matrices can also be prepared by this laboratory method as written.

20.3 The method as written does not incorporate the elements of EPA 901.1 t hat deal with instrument calibration and operation. These are found in AP -011.

20.4 Section 3 of the EPA referenced method is not included in this procedure. It is not the laboratory s responsibility to collect or preserve the samples.

Document Revision History

Revision	Effective Date	Changes From Previous Revision
17	10/23/13	• Document Revision History table implemented
18	10/23/14	• Reviewed: no changes necessary

Eberline Analytical Oak Ridge Laboratory Analytical Procedure

AP-002

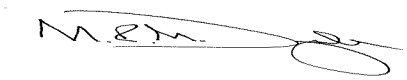
Sample Preparation (II): Digestion for Chemical Separations

AUTHORIZATION AND APPROVAL STATEMENT

This **Eberline Analytical** - Oak Ridge Laboratory, Analytical Procedure, Sample Preparation (II): Digestion for Chemical Separations is authorized and approved in its entirety by:

Saba Arnold Seaver
Quality Assurance Manager

Date: October 29, 2014



Michael R. McDougall
Laboratory Manager

Date: October 29, 2014

1.0 SCOPE, PURPOSE, AND APPLICABLE MARTICES

1.1 This procedure provides a method for the digestion of samples using mixed acids prior to elemental specific separations. The method also includes preparation of water samples or other matrices where direct co-precipitations or other preparation chemistry is conducted.

1.2 This method is applicable to all normal samples includi ng most water, soil, and solid sample.

1.3 This procedure includes elements of several EPA procedures including EPA 903.0 and 904.0, 907.0 and 908.0. It does not contain the entire EPA procedures. For purposes of organization, the parts included are limited to the chemical preparation steps of the listed EPA procedures.

2.0 DETECTION LIMITS

This section is not applicable. Please refer to specific counting methods.

3.0 SUMMARY OF METHOD

3.1 Samples routinely consist of water, soil or solid, air filters, vegetaton, or other matrices that may require preparation prior to chemical separation for specific analytes. Solid samples will routinely require digestion using mixed acids after initial sample preparation as described in this procedure. Additional preparations including selective co-precipitations and dilutions are routinely requiredSpecific digestion preparations may be required and in these cases directions will be provided by the Laboratory Manager.

3.2 Sample analyses almost always require the use of a radiometric tracer or elemental carrier for quantification of yield throughout the chemical separation process. Addition of these tracers , carriers and appropriate laboratory control samples are essential for determination of results and demonstration that quality control requirements are adhered to throughout the analytical process. The tracer shall be added to the sample after sub-sampling if required, but before any chemical treatment.

3.3 Samples are digested using mixed acids, selective precipitated and diluted as described herein. Samples may also require special cleanup chemistry as needed on a case-by-case basis. Analytical matrix modifications such as oxidation and reduction reactions are also detailed within this procedure.

Note
If analyzing for Americium/Curium, use the preparation described in AP -014

4.0 DEFINITIONS

MSDS Material Safety Data Sheets

NIST National Institute of Standards and Technology

LCS Laboratory Control Sample

MS Matrix Spike

TSS Total Suspended Solids, (Referring to the suspended solids fraction of a water sample)

TDS Total Dissolved Solids, (Referring to the dissolved solids fraction of a water sample)

Oxidation or Reduction – A chemical reaction in which one component loses electrons or is oxidized and another gains electrons or is reduced.

5.0 INTERFERENCES

5.1 This procedure is structured to be applicable to a wide array of sample matrices. Unique samples will be dealt with as directed by the Laboratory Manager.

5.2 Samples containing Barium exceeding 10.0 mg per aliquot will interfere with the resolution of the Alpha spectra for Ra-226 determinations.

5.3 Yttrium-90 is not separated from Actinium-228 using this preparation method. Samples that are known to contain Sr-90 cannot be accurately prepared for Radium-228 analyses by the EPA 904.0 method which this method references. In these instances the laboratory will use the EiChroM Ra-01 method.

5.4 The presence of elevated levels of radionuclides in client samples, including the isotopes of interest, can lead to analytical problems including, but not limited to, low chemical recovery, spectral degradation, interference from other nuclides, and high method detection limits. In these cases it may be necessary to use an aliquot which is significantly lower than those recommended in this procedure. In these instances the Laboratory Manager will provide specific instructions.

6.0 SAFETY

Laboratory chemical and general safety shall be conducted as required within *Eberline Analytical – Oak Ridge Laboratory Chemical Hygiene/Health & Safety Plan , Latest Version*

Laboratory radiation safety shall be conducted as required within *Eberline Analytical – Radiation Protection Plan and Attachments , Latest Version*

Waste management and sample return shall be conducted as required within *Eberline Analytical – Waste Management Plan , Latest Version*

6.1 Housekeeping

6.2 All work areas shall be kept as clean as possible at all times and the entire work area shall be cleaned at the conclusion of each shift.

6.3 Minimize the amount of lose paper that is present in the work space.

6.4 Promptly clean any spills that occur using the guidance contained in the Emergency Action Plan, Spill Response Procedure and support of the Radiation Safety Officer and Health and Safety Manager if necessary.

6.5 Clearly label all sample containers (beakers, bottles, c-tubes etc.) with the work order number, analysis fraction, and analyte identification information such as Total Sr , Iso-U , or some other recognizable wording.

6.6 Any labels that identify the hazards associated with a particular container at the time of receipt will remain affixed to that container AND to ALL subsequent sub sampling from, and disposal of that container.

6.7 Dispose of all waste in the appropriate containers as directed by the Waste Management Plan.

6.8 Dispose non-rad waste in appropriate containers, DO NOT PUT NON-RAD WASTE INTO RAD WASTE CONTAINERS.

6.9 Personal protective equipment for this procedure shall consist of a lab coat or protective apron, safety glasses or goggles and chemical resistant laboratory gloves.

7.0 EQUIPMENT AND SUPPLIES

7.1 Assorted pipettes

7.2 Magnetic stir bars

7.3 Magnetic stirrer-hot plate

7.4 Balance (0-3600.00 grams capacity)

7.5 Centrifuge

7.6 Hot plate

7.7 Vortex stirrer

7.8 Muffle furnace

7.9 Heat lamp

7.10 Analytical balance

7.11 Auto Dispensing Pipettes (50 µL, 100 µL, 200 µL, 250 µL, 500 µL, and 1000 µL)

7.12 Assorted laboratory glassware

7.13 50- and 250-ml centrifuge tubes

7.14 pH indicator strips, pH 0-6, 7-14

7.15 pH Meter

7.16 Air/gas Bunsen burner

7.17 Carborundum & Glass Boiling Chips

7.18 Gelman 47-mm filter holder designed to retain filtrate

7.19 Gelman 47-mm 0.45-µm filters

7.20 The laboratory may use pre-cleaned disposable plastic lab ware as appropriate and applicable to this or any other analytical procedure. Disposable plastic ware will be disposed of in the appropriate waste container after use.

8.0 REAGENTS AND STANDARDS

8.1 Ammonium hydroxide, (NH_4OH), concentrated, reagent grade

8.2 10M Ammonium hydroxide, (NH_4OH)

8.3 30% Hydrogen peroxide, (H_2O_2), reagent grade

8.4 Hydrofluoric acid, (HF), 48%, reagent grade

8.5 Nitric acid, (HNO_3), 70%, 16 Normal, reagent grade

8.6 Hydrochloric acid, (HCl), 36%, 12 Normal, reagent grade

8.7 Perchloric acid, ($HClO_4$), 70%, 12 Normal, reagent grade

8.8 Barium carrier (1 mg/ml): Dissolve 1.9 g of Barium nitrate [$Ba(NO_3)_2$] in deionized water and dilute to 1 liter with deionized water in a volumetric flask.

8.9 Ammonium sulfate (200 mg/ml): Dissolve 200 g of Ammonium sulfate [$(NH_4)_2SO_4$] in 750 ml of deionized water. Bring up to 1-liter total volume.

8.10 Lead carrier (166 ml): Dissolve 265.3 g of Pb $(NO_3)_2$ in deionized water and dilute to 1 liter with deionized water in a graduated cylinder.

8.11 Sulfuric acid, reagent grade, (H_2SO_4), 95% to 98%, 36 Normal

8.12 Lead carrier (40 mg/ml): Dissolve 63.89 grams of Lead nitrate [Pb $(NO_3)_2$] in 0.0.5N HNO_3. Add 0.0.5N HNO_3 to reach a total volume of 1 liter and mix again.

8.13 Potassium sulfate, (K_2SO_4), crystals, reagent grade

8.14 8N HNO_3: Add 500 ml concentrated Nitric acid to 400 ml of deionized water. Add deionized water to reach a total volume of 1 liter and mix again.

8.15 Potassium Hydroxide, 10M (KOH) Add 560 grams of KOH to 1000 ml of deionized H_2O. Mix until in solution.

8.16 Anhydrous Potassium fluoride, (KF), reagent grade

8.17 Anhydrous Potassium bifluoride, (KHF_2), reagent grade

8.18 Potassium nitrate, (KNO_3), reagent grade

8.19 Anhydrous Sodium sulfate, (Na_2SO_4), reagent grade

8.20 Potassium persulfate, ($K_2S_2O_5$), reagent grade

8.21 Fusion Flux: 100 g of KF added to 70 g of KHF_2 and 2 g of KNO_3 Blend thoroughly using jar mill, mortar and pestle or similar method to insure that all chunks of material are broken up and the reagents are thoroughly mixed.

8.22 Tellurium oxide, 0.625%: In 100 ml volumetric flask place 25 ml of concentrated Hydrochloric acid. Add 0.625 grams of Tellurium oxide and swirl until dissolved. Slowly add water until volume reaches 100 ml. Stopper and invert several times to mix thoroughly. Transfer to a labeled bottle.

8.23 Potassium persulfate solution, 25%: Place 25 grams of $K_2S_2O_5$ in a 100 ml volumetric flask and add water to a total volume of 100

8.24 Safranin 0, 0.1%: add 0.1 g Safranin 0 to 100 ml of deionized water and swirl to mix.

8.25 0.25 M EDTA (Ethylenediaminetetraacetic acid): Carefully dissolve 20 grams of Sodium hydroxide in 800 ml of deionized water, then slowly add 93 grams of EDTA. Stir and dilute to 1 liter with deionized water.

8.26 Phenolphthalein: Dissolve 0.1 g of phenolphthalein in 60 mls of reagent Alcohol and dilute to 100 mls with deionized water.

8.27 Reagent Alcohol

8.28 Ba carrier (50 mg/ml): Dissolve 95.0 g Barium nitrate [$Ba(NO_3)_2$] and dilute to 1 liter with DI water in a volumetric flask.

8.29 Thioacetamide solution. Weigh out approximately 4 grams of Thioacetamide [CH3CSNH2] and dilute to 100 milliliters.

8.30 Barium-133 standard solution. NIST traceable solution prepared according to MP -009.

8.31 Radium-226 standard solution. NIST traceable solution prepared according to MP -009.

8.32 Radium-228 standard solution. NIST traceable solution prepared a ccording to MP-009.

8.33 Thorium-232/228 standard solution. NIST traceable solution prepared according to MP -009.

8.34 Thorium-230 standard solution. NIST traceable solution prepared according to MP -009.

8.35 Thorium-229 standard solution. NIST traceable solution prep ared according to MP-009.

8.36 Uranium-238/235/234 standard solution. NIST traceable solution prepared according to MP -009.

8.37 Uranium-232 standard solution. NIST traceable solution prepared according to MP -009.

8.38 Plutonium-238 standard solution. NIST traceable s olution prepared according to MP -009.

8.39 Plutonium-239 standard solution. NIST traceable solution prepared according to MP -009.

8.40 Plutonium-242 standard solution. NIST traceable solution prepared according to MP -009.

8.41 Americium-241 standard solution. NIST tra ceable solution prepared according to MP -009.

8.42 Americium-243 standard solution. NIST traceable solution prepared according to MP -009.

8.43 Curium-243 standard solution. NIST traceable solution prepared according to MP -009.

8.44 Neptunium-237 standard solution. NIST traceable solution prepared according to MP -009.

8.45 Americium-243 standard solution. NIST traceable solution prepared according to MP -009.

9.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

9.1 Sample collection and preservation is not the responsibility of the labo ratory and is not applicable to this procedure. Upon receipt of water samples, the laboratory may preserve/pH adjust the samples depending on the composition of the sample and the requested analysis.

9.2 Unless otherwise directed by the client, after receipt, all soil, solid, water, and vegetation samples will be segregated according to preliminary activity scans and stored in a secure, climate controlled location. Tissue sample will be stored in a freezer prior to analysis.

10.0 QUALITY CONTROL

10.1 One Laboratory Control Sample (LCS) shall be analyzed with every 20 samples. The LCS will be prepared and analyzed the same way and along with the analysis batch for the same analytical parameter.

10.2 One analysis blank shall be analyzed with every 20 samples. If there are less than 20 samples per analysis batch, then one blank per batch shall be analyzed.

10.3 A minimum of one or a designated number of client samples shall be duplicated with every 20 samples (one sample for every 10 client samples will be duplicated for RCRA or SW846 analyses). If there are less than 20 samples per analysis batch, then a minimum of one or a sufficient number of duplicates to meet client criteria shall be analyzed per analytical batch. Where the matrix type, limited sample volume or other speci al considerations preclude this as a viable option, a replicate analysis will be used for QC evaluation

10.4 If requested by a client, a matrix spike composed of a sample spiked with a standard containing at least one of the isotopes in question (NIST traceable or equivalent) shall be run with each batch.

10.5 Other client specific requirements or criteria may supersede these requirements.

11.0 CALIBRATION AND STANDARDIZATION

11.1 There are no standardized carriers used for this procedure.

11.2 The calibration of the alpha, beta, and gamma detectors are covered in procedure(s) AP-011, AP-018, AP-023, and AP-029.

11.3 The dilution of NIST traceable standard solutions is covered in procedure MP -009.

11.4 The calibration check for balances is covered in procedure MP -010.

11.5 The use, maintenance, and volume verification of the mechanical pipettes is covered in MP -025

12.0 PROCEDURE

12.1 Preparation of sediment -Free Water Samples for Radium Analysis (Radium -226, Radium-228, and Total Radium)

12.1.1 Label the appropriate number of beakers for the client samples. Add Ba-133 tracers to the beakers as required including the laboratory control sample and blank beakers. Record

the weight of the Ba-133 tracer and record within LIMS in Laboratory Technician Functions, Tracer Data function. Record the weights of the Ra-226 standard and Ra-228 also, if requested, within the appropriate section of the LIMS. If a matrix spike is required, directions will be provided within the analytical sub file for this requirement. This sample will be designated as MS for matrix spike and MSO for the non-spiked sample within the LIMS. If the same sample is duplicated and matrix spiked, the non-spiked sample would be designated an DMSO within the LIMS. Activity levels for the matrix spike should be equal to the laboratory control sample unless otherwise directed by the Laboratory Manager. Activity levels for the matrix spike should be equal to the laboratory control sample unless otherwise directed by the Laboratory Manager. Matrix spike additions may need to be significantly higher than the laboratory control sample if samples contain positive activity. The Laboratory Manager will provide specific directions in the event samples are positive. In general, laboratory control samples will be at a level that does not exceed 10 times the laboratory advertised method detection limits. This level of activity may also be changes at the discretion of the Laboratory Manager.

12.1.2 Open the LIMS, Laboratory Technician Functions, Aliquot/Dilution Data section and enter the sample identification into the LIMS system. Transfer the required sample volumes into their corresponding beaker and document all aliquots. For the laboratory control sample, an aliquot of one shall always be used. For the blank, the aliquot will be one unless otherwise directed in the analytical sub file. .

12.1.3 Adjust the sample pH, drop-wise with concentrated Ammonium hydroxide or Nitric acid to a final pH of 2.8 to 3.0.

12.1.4 Add 3 ml 1.0 mg/ml Barium carrier.

12.1.5 Add 1 ml 166 mg/ml Pb carrier.

12.1.6 Add 20 ml of 200 mg/ml $(NH_4)_2 SO_4$. In the event that the sample appears to contain excessive sulfate forming elements, additional $(NH_4)_2 SO_4$ may be required. If sample has copious sulfate precipitates, see the Laboratory Manager.

12.1.7 Stir for approximately 10 - 20 minutes.

12.1.8 Remove stir bars and allow to sit until the precipitate has completely settled, (typically 2 to 4 hours).

12.1.9 Decant the supernatant to the waste disposal (hot) sink unless otherwise directed by laboratory management

12.1.10 Transfer the precipitate into 50 ml centrifuge tube using DI H_2O wash. Centrifuge for 5 minutes at maximum rpm setting.

12.1.11 Pour off the supernatant to the hot sink.

12.1.12 Document analytical steps within the analytical logbook, sign and place a copy of such laboratory technician notes with the analytical sub file.

12.1.13 Complete internal chain of custody information and transfer samples to separations as appropriate.

12.2 Preparation of soils and Solid Matrices for Radium Analysis: Open the LIMS Laboratory Technician Functions, Aliquot/Dilution Data section and enter the sample identification into the LIMS system. Place approximately 1 to 5 grams of soil or solid matrix as appropriate into a 250 ml beaker.

NOTE

Documentation of soil or solid sample aliquots shall be conducted using the balance interface in order to avoid transcription errors.

12.2.1 For each batch of samples, a laboratory control sample, blank and duplicate/replicate shall be analyzed as appropriate. For the laboratory control sample, an aliquot of 1 shall be used. For the blank, the aliquot will be 1 unless otherwise directed by the analytical sub file.

12.2.2 Add Barium-133 tracer to all samples including the laboratory control sample and blank and record within LIMS in Laboratory Technician Functions, Tracer Data function. Record weight of Ra-226 standard and if Ra-228 analysis is also requested, record weight of this standard also within this LIMS function. If a matrix spike is required, directions will be provided within the analytical sub file for this requirement. This sample will be designated as MS for matrix spike and MSO for the non-spiked sample within the LIMS. If the same sample is duplicated and matrix spiked, the non-spiked sample would be designated an DMSO within the LIMS. spike within the LIMS. Activity levels for the matrix spike should be equal to the laboratory control sample unless otherwise directed by the Laboratory Manager.

12.2.3 Wet sample with DI water and add approximately 3 to 5 ml concentrated HF (additional volume may be required depending upon sample matrix) and heat to dryness. Cool. Note: Samples may react vigorously if HF is added to the dry sample. It is imperative that water be added prior to HF additions. Water acts as a buffer to slow any initial reactions. (this step may be omitted at the direction of the Laboratory Manager).

12.2.4 Add 10 ml of 70 %HNO3.

12.2.5 Add 15 ml of 70% $HClO_4$. (NEVER ADD TO SAMPLE THAT CONTAINS ORGANIC MATTER)

12.2.6 Add 1 ml concentrated H_2SO_4. Place samples on hotplate. Heat the samples to fuming H2SO4. These are typically thick hanging white fumes. All other acids will be removed by this process. Continue heating until dry. Remove from hot plate and allow to cool to room temperature.

NOTE

Additions of H_2SO_4. will cause samples to boil in the presence of water. Additions of H_2SO_4. shall be slowly as to mix with other acids and water present and not cause excessive reaction or boiling. Never add water to concentrated H_2SO_4. Always add H_2SO_4 to water or acids.

12.2.7 After digestion is completed, add approximately 150 ml of DI water and measure the pH using meter or strips. Adjust the pH to be between 2.8-3.0. Adjust pH dropwise with concentrated Ammonium hydroxide or Nitric acid.

12.2.8 Heat to near boiling and add approximately 5.0 g of K_2SO_4 and small additions of H_2O_2. Stir if necessary to dissolve the solid Potassium Sulphate.

12.2.9 Add 3 ml of 1.0 mg/ml Barium carrier.

12.2.10 Add approximately 3 ml of 40 mg/ml Lead carrier.

12.2.11 Stir near boiling solution for approximately 10 - 15 minutes.

12.2.12 Remove the stir bars and allow sample to sit until the precipitate has completely settled, (approximately four hours).

12.2.13 If Uranium is needed, save the supernatant (see Uranium Notes Below). Otherwise, decant the supernatant and discard to the hot sink.

12.2.14 Transfer precipitate into 50-ml centrifuge tube using deionized water wash. Centrifuge for 5 minutes at maximum.

12.2.15 Save supernatant and wash solution if Uranium is needed (see Uranium Notes Below). Otherwise, pour off supernatant and discard to the hot sink.

12.2.16 Add 20 ml of 0.25M EDTA and two drops phenolphthalein indicator. Indicator is to insure proper pH, (PINK). If solution color is not pink, add 10M NH_4OH dropwise until pink.

12.2.17 Vortex the sample and place in hot H2O bath for approximately 10 minutes, (do not overcook, since evolution of NH_4 will occur and pH may be lowered). Centrifuge and retain the supernatant. Discard the precipitate to the Dry Active Waste Receptacle, unless Thorium is needed.

12.2.18 Document the analytical steps within the analyst notes or in the electronic notes section of the LIMS.

12.2.19 Complete internal chain of custody information and transfer samples to s eparations as appropriate.

12.3 Preparation of soils / Solids for Radium Analysis - Matrix Interferences

12.3.1 Open the LIMS Laboratory Technician Functions, Aliquot Dilution Data and enter the sample identification into the LIMS system. Add approximately 1 gram of the sample to a 50 ml centrifuge tube. (The spike and blank will consist of 20 mls of 0.25M EDTA and 3 milliliters of 1 mg/ml Barium carrier with the appropriate amount of standard solutions and tracers). The weight for both the Laboratory Control Standar d and the Blank will be 1 unless otherwise directed.

12.3.2 Record weight of Ba-133 tracer and add to all samples as required including the laboratory control sample and blank and record within LIMS in Laboratory Technician Functions, Tracer Data function.

12.3.3 Record weight of Ra-226 standard and if Ra-228 analysis is also requested, record weight of this standard also within this LIMS function. If a matrix spike is required, directions will be provided within the analytical sub file for this requirement. This sample will be designated as MS for matrix spike and MSO for the non-spiked sample within the LIMS. If the same sample is duplicated and matrix spiked, the non-spiked sample would be designated an DMSO within the LIMS. Activity levels for the matrix spike should be equal to the laboratory control sample unless otherwise directed by the Laboratory Manager. Matrix spike additions may need to be significantly higher than the laboratory control sample if

samples contain positive activity. The Laboratory Manager will provide specific directions in the event samples are positive. In general, laboratory control samples will be at a level that does not exceed 10 times the laboratory advertised method detection limits. This level of activity may also be changes at the discretion of the Laboratory Manager.

12.3.4 Add 20 ml of 0.25M EDTA, 1 drop of 0.1% phenolphthalein, 3 drops of concentrated NH_4OH and an additional two milliliters of 1 mg/ml Barium carrier. Vortex and heat in a hot water bath for 15 minutes continuously adding NH_4OH as needed in order to maintain a pink color. Be sure not to have the centrifuge lids on too tightly while the samples are sitting in the water bath. Vortex and centrifuge the samples. Decant the supernatant into a clean centrifuge tube. Send to separations for additional chemistry.

12.3.5 Document analytical steps within the electronic analyst notes, sign and place a copy of such laboratory technician notes with the analytical sub file.

12.3.6 Complete internal chain of custody information and transfer samples to separations as appropriate.

12.4 Preparation of Water samples for Thorium

12.4.1 Obtain a sufficient number of beakers for all of the samples in the work order. Use appropriate sized beakers for designated aliquots.

12.4.2 Open the LIMS, Laboratory Technician Functions: Aliquot/Dilution Data section and enter the sample identification into the LIMS system. Transfer the required sample volumes into their corresponding beakers and document all aliquots within the LIMS. For the laboratory control sample, an aliquot of one shall always be used unless otherwise directed on the aliquot sheet. For the blank, the aliquot will be one unless otherwise directed on the aliquot sheet.

12.4.3 Add appropriate tracers to all sample beakers including the laboratory control sample, blank and a matrix spike if required and using the LIMS section, Laboratory Technician Functions, Tracer Data function. Record the weight of Thorium-228, Thorium-230, Thorium-232 standards, and the Thorium-229 tracer activity within this LIMS function.

12.4.4 If a matrix spike is required, directions will be provided within the analytical sub file for this requirement. This sample will be designated as MS for matrix spike matrix spike and MSO for the non-spiked sample within the LIMS. If the same sample is duplicated and matrix spiked, the non-spiked sample would be designated an DMSO within the LIMS. Activity levels for the matrix spike should be equal to the laboratory control sample unless otherwise directed by the Laboratory Manager. Activity levels for the matrix spike should be equal to the laboratory control sample unless otherwise directed by the Laboratory Manager. Matrix spike additions may need to be significantly higher than the laboratory control sample if samples contain positive activity. The Laboratory Manager will provide specific directions in the event samples are positive. In general, laboratory control samples will be at a level that does not exceed 10 times the laboratory advertised method detection limits. This level of activity may also be changes at the discretion of the Laboratory Manager.

.

NOTE
For Thorium analyses, where samples are suspected to contain elevated

Uranium activity, add three drops of 0.1%, Safranin-O solution. If the solution is clear, the sample is oxidized. Proceed to Section 12.6.14. If the samples are still pink, add H_2O_2 dropwise until the sample becomes clear. If there has been no color change after the addition of a few milliliters of Hydrogen Peroxide, contact the Laboratory Manager for directions. It is essential that samples be completely oxidized when Uranium is present. Uranium will not precipitate as a sulfate in an oxidized state.

12.4.5 Place the samples on stirring hot plates with stir bars in sample beakers and heat to boiling. Add ~5 grams of Potassium sulfate (K_2SO_4) and let the sample stir until the solids are dissolved.

12.4.6 Add 1-ml of 50 mg/ml Ba carrier. Continue to boil, taking care to prevent boil over and bumping of sample and stirring for 10 minutes.

12.4.7 Turn off heat, remove stir bars, and let samples cool for 15 to 30 minutes. Let samples settle for four hours or as appropriate until precipitates have completely settled.

12.4.8 After all sample precipitates have settled, decant supernatant and discard into the waste disposal sink.

12.4.9 Transfer precipitate to clean 50 ml centrifuge tube with D.I. H_2O and centrifuge. Discard the supernatant into the hot sink. Repeat this step if necessary to transfer all the precipitate into the centrifuge tube.

12.4.10 Add 20 ml of 0.25M EDTA and vortex the samples. Place the centrifuge-tubes in a hot water bath for approximately 10 minutes. Add 0.20 ml of 10M KOH and 0.25 ml of Iron carrier. Return centrifuge tubes to hot water bath for another ten minutes.

12.4.11 Centrifuge samples in C-tubes for 5 minutes, then discard the 0.25M EDTA supernatant in the hot sink.

12.4.12 Add 30 ml of 8N Nitric acid (HNO_3) and vortex. Put samples back in the water bath until samples turn clear.

12.4.13 Document analytical steps within the analytical logbook, sign and place a copy of such laboratory technician notes with the analytical sub file.

12.4.14 Complete internal chain of custody information and transfer samples to separations as appropriate.

12.5 Preparation of Water Samples for Uranium and Plutonium

12.5.1 Prepare a separate set of sample beakers for each of the following radionuclides unless otherwise directed by the Laboratory Manager: Uranium, and Plutonium. In certain instances sequential chemistry may be required. In these instances, the Laboratory Manger will provide work order specific instructions.

12.5.2 Open the LIMS, Laboratory Technician Functions; Aliquot/Dilution Data section and enter the sample identification into the LIMS system. Using a graduated cylinder, transfer the required sample volumes, typically 500 ml, into their corresponding glass beaker and document all aliquots.

12.5.3 For the laboratory control sample, an aliquot of 1.0 shall always be used. For the blank, the aliquot will be 1.0 unless otherwise directed by a member of the laboratory management. If water samples contain a high TSS content, proceed to Section 12.6 .

12.5.4 Add the tracers, carriers and standard solutions to the beakers as required including the laboratory control sample and blank and record the weight of standards used within the LIMS in Laboratory Technician Functions, Tracer Data function.

12.5.5 If a matrix spike is required, directions will be provided within the analytical sub file for this requirement. This sample will be designated as MS for matrix spike within the LIMS. Activity levels for the matrix spike should be equal to the laboratory control s ample unless otherwise directed by the Laboratory Manager or.

12.5.6 If the samples are to be analyzed for Plutonium and Uranium, heat to near

12.5.7 Complete internal chain of custody information and transfer samples to separations as appropriate.

NOTE

Watch heat leve l and add boiling chips to prevent boil over or bumping of sample. Never add boiling chips or glass beads after that sample has been heated.

12.6 Preparation of High TSS and Solid or Soil Samples for Uranium, Thorium, or Plutonium Analysis

12.6.1 Soil samples and wate r samples that contain a high TSS content may require special preparation for mixed acid dissolution prior to specific separation chemistry for Uranium, Thorium, and Plutonium. In these instances, aliquot size is frequently dependent upon the TSS or activity content within the sample. Determination of aliquot size to be used shall be made with the assistance of the Laboratory Manager. Additionally, in the case of samples that are known or suspected to have significantly elevated levels of activity, a reduce d aliquot of the sample may be digested and then diluted to a fixed volume after which aliquots will be removed as necessary for the analysis of individual radionuclides.

12.6.2 Aliquot the following radionuclides separately unless otherwise directed by the Labor atory Manager: Uranium, and Plutonium. In certain instances sequential chemistry may be required. In these instances, the Laboratory Man ager will provide work order specific instructions.

12.6.3 Open the LIMS Laboratory Technician Functions, Aliquot/Dilution Dat a section and enter the sample identification into the LIMS system. For water samples, use a graduated cylinder and aliquot 500 ml of samples into appropriate size beakers or sample specific aliquots volumes as directed by the Laboratory Manger. For soils use 1.0 gram or sample specific aliquots as directed by the Laboratory Manger. Transfer the required sample aliquots into suitable sized beakers and document all aliquots.

12.6.4 Add the carriers, tracers, and standard solutions to the empty beakers. Record the weights of the standard and tracer solutions used within the LIMS in Laboratory Technician Functions, Tracer Data function. Record weight of standards added within LIMS in Laboratory Technician Functions, Tracer Data function.

12.6.5 If a matrix spike is required, directions will be provided within the analytical sub file for this requirement. This sample will be designated as MS for matrix spike within the LIMS.

Activity levels for the matrix spike should be equal to the laboratory control sample unless otherwise directed by the Laboratory Manager.

12.6.6 Water samples that contain a high TSS content shall be heated to near dryness.

12.6.7 If samples are dry, wet the sample with DI water. For Thorium, go to 12. 6.8. For Uranium and Plutonium, add approximately 3 to 5 ml concentrated HF (additional volume may be required depending upon sample matrix) and heat to near dryness. Cool. Note: Samples may react vigorously if HF is added to the dry sample. It is imperative that water be added prior to HF additions. Water acts as a buffer to slow any initial reactions.

12.6.8 Add 10 ml of concentrated Nitric acid (HNO_3) and 15 ml concentrated Perchloric acid ($HClO_4$) to all sample beakers. Samples that contain any organic material must be muffled prior to the addition of any oxidizing acids.

NOTE
Additions of H_2SO_4 will cause samples to boil in the presence of water. Additions of H_2SO_4 shall be done cautiously allowing it to slowly mix with other acids and water present. Never add water to concentrated H_2SO_4. Always add H_2SO_4 to water or acids.

12.6.9 For samples requiring Uranium, Thorium, and Plutonium analyses, add 1 or 2 ml concentrated Sulfuric acid (H_2SO_4). Place samples on hotplate and heat to dryness.

12.6.10 Remove from hot plate and allow the samples to cool to room temperature.

12.6.11 If Uranium is potentially in the Thorium analysis aliquot, or if a matrix is problematic, add approximately 150 ml of DI water and heat to near boiling. Where samples are suspected to contain elevated Uranium activity, add three drops of 0.1%, Safranin -O solution. If the solution is clear, the sample is oxidized. If the samples are still pink, add H_2O_2 dropwise until the sample becomes clear. It is essential that samples be completely oxidized when Uranium is present. Uranium will not precipitate as a sulfate it the sample is in an oxidized state.

12.6.12 For Thorium, place samples on stirring hot plates with stir bars in sample beakers, and heat to boiling. Add approximately 5 grams of Potassium sulfate (K_2SO_4) and let it dissolve. Add 1-ml of 50 mg/ml Ba carrier. Continue t o boil, taking care to prevent boil over and bumping of sample and stirring for 10 minutes.

12.6.13 Turn off heat, remove stir bars, and let samples cool for 15 to 30 minutes. Let samples settle for 4 hours or as appropriate until precipitates have completely sett led.

12.6.14 After all sample precipitates have settled, decant supernate and discard into the waste disposal sink.

12.6.15 Transfer the precipitate to clean 50 ml centrifuge tubes with D.I. H_2O and centrifuge. Discard the supernate into the hot sink. Repeat this step if necessary to transfer all the precipitate into the centrifuge tube.

12.6.16 Add 20 ml of 0.25M EDTA and vortex the samples. Place the c -tubes in a hot water bath for approximately 10 minutes. Add 0.20 ml of 10M KOH and 0.25 ml of Iron carrier. Return centrifuge tubes to hot water bath for another ten minutes.

12.6.17 Centrifuge samples in C-tubes for 5 minutes, then discard the 0.25M EDTA supernate in the hot sink.

12.6.18 Add 30 ml of 8N Nitric acid (HNO_3) and vortex, place samples back into hot waterbath until samples turn clear

12.6.19 Document analytical steps within the electronic analyst notes, sign and place a copy of such laboratory technician notes with the analytical sub folder.

12.6.20 Complete internal chain of custody information and transfer samples to separations as appropriate.

12.7 Preparation of Air filter Samples for Alpha/Beta Emitting Radionuclides

12.7.1 Upon receipt-and depending on the filter media, transfer the sample to a Teflon or glass beaker as appropriate.

a) For glass fiber filters, place filters into a Teflon beaker, wet the sa mples and add concentrated Hydrofluoric acid slowly to the filters until dissolved. Evaporate to dryness and transfer sample to a glass beaker. Use concentrated HNO_3 and wash Teflon beaker three times.

b) For cellulose nitrate or other media, place sample s into a glass beaker, add concentrated HNO_3 until filter is in solution.

c) For samples that require the analysis of volatile radionuclides, see the for specific digestion instructions.

12.7.2 Air filter samples that are to be analyzed for non-volatiles. See the Laboratory Manager for specific digestion instructions.

12.7.3 Upon completion of digestion, centrifuge if necessary and where multiple analyses of the sample are required, dilute the samples to an appropriate volume for allocation of aliquots as required for all analytes.

12.7.4 Take appropriate aliquots from dilution for each analysis, add analytical tracers and prepare the laboratory control sample for each analyte. Conduct analytical steps as necessary as noted previously for each specific analyte.

12.7.5 Where multiple analyses are required for ^{226}Ra, ^{228}Ra, Isotopic Thorium and Isotopic Uranium, and sequential separation chemistry is employed, add tracers for all of the analyses as required.

12.7.6 Add approximately 100 ml of DI water as appropriate, adjust pH to 2.8 to 3.0, add approximately 5 ml of H_2O_2 to thoroughly oxidize, bring to boil and conduct steps in 12.2.10 through 12.2.14.

12.7.7 After initial $PbSO_4$ and $BaSO_4$ precipitations, (save the supernatant for Isotopic Uranium). Place supernatant into an appropriate sized beaker, dry down and send to separations. (The supernatant contains Uranium)

12.7.8 To the $PbSO_4$ and $BaSO_4$, (the precipitate contains ^{226}Ra, ^{228}Ra and Thorium). Add 20 ml of 0.25M EDTA and vortex the samples. Place the c-tubes in a hot water bath for

approximately 10 minutes. Add 0.20 ml of 10M KOH and 0.25 ml of Iron carrier. Return centrifuge tubes to a hot water bath for another ten minutes.

12.7.9 Centrifuge centrifuge-tubes for 5 minutes, then save the supernatant, and submit for Radium analyses. Add 30 ml of 8N Nitric acid (HNO_3) to the precipitate and vortex out samples back into the hot water bath until clear.

12.7.10 Print the analysis sheet from the LIMS within, Laboratory Technician Functions, Print Analysis Sheet and place in analytical sub file. Complete internal chain of custody information and transfer samples to separations as appropriate.

12.8 Vegetation /Tissue Samples - Non-Volatile └/ß Emitting Radiouclides

12.8.1 After receiving the ashed sample from rough sample preparation, and upon supervisory direction, either go to Step 12.6 or: if analyzed for Neptunium, go to AP -010; if analyzed for Americium/Curium use AP -014 12.2; or if necessary (not adequately ashed) and upon direction, weigh an appropriate aliquot (typically 1 gram) and leach in mixture of concentrated Nitric acid and concentrated Hydrochloric acid. Follow procedures depending on analyte.

12.8.2 Spike and Trace the samples upon direction from the Laboratory Manager. In some cases the sample will be diluted and aliquots taken from the dilution.

12.9 Sample Preparation by Leaching (Metallic, Special Solids)

12.9.1 Open the LIMS Laboratory Technician Functions: Aliquot/Dilution Data section and scan each sample container to enter the sample identification into the LIMS system. Transfer the required sample aliquots into a suitable sized glass beaker and document all aliquots. Typically, one gram can be used as the sample aliquot although the Laboratory Manager should always verify this. For the Laboratory Control Standard an aliquot of 1.0 shall be used. For the blank, the aliquot will be one unless otherwise directed by the laboratory management.

12.9.2 See the Laboratory Manager for specific digestion instructions.

12.9.3 Separate the residual sample material and the acid leaching solution if present, (dependent upon the type of leach or digestion used). If present, save the acid leaching solution for sample analysis (as per the appropriate section in this procedure). In some cases the sample will be diluted and aliquots taken from the dilution. In the case of total dissolution, no solution may be present. In these cases sample will be directly transferred to chemistry separations as appropriate.

12.10 Sample Digestion by Fusion

12.10.1 Place approximately one gram of solids in a Platinum dish and add two to three milliliters of concentrated Nitric acid. After all evolution of gas ceases, add any tracers or spikes to the dish and place either on a low temperature hotplate or under a heat lamp to evaporate all liquids.

12.10.2 After all water has evaporated, break up the resulting cake with the end of a glass rod. Add 3.5 grams of fusion flux, (See 8.21) and thoroughly mix with the sample using the end of a glass rod.

12.10.3 Place the Platinum dish in the wire triangle over blast burner and heat with hottest possible flame until a clear fusion is obtained. Remove the dish from the flame and allow to cool to room temperature. When removing the dish, do not grasp the edge of the dish with the t ips of the tongs. The dish should be gently grasped around the bottom using the wide part of the tongs.

12.10.4 While the dish is cooling, combine 70 ml of DI water and 10 ml of concentrated HCl in a 250-ml beaker and place on a hot plate. This solution should be heated to near boiling when used in Section 12.11.11.

12.10.5 Add 4 ml of concentrated Sulfuric acid to the dish and heat on a hotplate set on medium heat until the fusion cake is completely dissolved. This may require some gentle stirring with the glass rod and an additional milliliter or two of Sulfuric acid. **This reaction can be quite vigorous.** Be sure to avoid spattering Sulfuric acid and do not let the samples froth over.

12.10.6 After the frothing has stopped, add 0.5 gram of KF to the mixture and allow to heat on th e hot plate until any further frothing has stopped.

12.10.7 Add 2 grams of anhydrous Na2SO4 to the mixture and return it to the wire triangle over the blast burner.

12.10.8 Heat slowly at first to drive off the water present in the Sulfuric acid and then increase heat until H_2SO_4 fumes are generated. Continue to heat the sample until a clear red -colored pyrosulfate fusion melt is obtained. It may be necessary to tilt the dish slightly to get all of the material in the dish into solution.

12.10.9 Remove the dish from the heat and a s the fusion melt cools, tilt the dish to deposit the cooling material on the sides of the dish in as thin a layer as possible. This will make it much easier to remove the sample later.

12.10.10 Once the dish has reached room temperature, gently flex the sides and rap the bottom of the dish on the stone counter top while covering the top of the dish with the palm to prevent spilling any bits that may fly out.

12.10.11 Carefully transfer the contents of the dish and the dish itself into the hot dilute HCl solution, prepared in Section 12.11.4. Any bits of material sticking to the dish should come off almost instantly. As the dish is removed, rinse with a small amount of distilled water.

12.10.12 Add two or three boiling chips, cover with a watch glass and boil until the last traces of the sulfate cake have been dissolved. This solution should be clear and colorless to dark yellow at this time. It is necessary to dissolve the cake as quickly as possible in order to prevent the formation of insoluble $CaSO_4$.

12.10.13 Once the cake is dissolved, set the beaker off the hotplate and allow to cool to near room temperature. Add 200 µl of 0.625% TeO_2 solution and 1 ml of 25% K_2S2O_4. Cover with a watch glass and return to the hotplate.

12.10.14 When the first bubbles from boiling break the surface, remove from the hotplate and set the solution aside until a black dispersion of Tellurium metal forms in the solution. Return to the hotplate and boil for about five minutes to flocculate the Tellurium and volatilize the excess sulfite.

12.10.15 Add 1 ml of additional TeO_2 solution and boil for one or two additional minutes to assure oxidation of the last traces of sulfates. Add two drops of 20% $TiCl_3$ to the boiling sample to reduce the last of the Tellurium and add four drops excess. Set the beaker off the hotplate for three minutes or so to retard the flocculating of the Tellurium.

12.10.16 Heat the solution back to boiling and filter through a 0.45 -micron filter in a Gelman filter holder and catch the filtrate in the lower chamber. Retain the filtrate in the lower chamber for further analysis for any non-transition metal.

12.11 Optional Clean-Up Steps

12.11.1 Calcium Phosphate Precipitation

12.11.2 In many cases a Calcium phosphate precipitate step will be required to concentrate the actinides in the sample and help separate these species of interest from poten tial contaminants in the sample.

12.11.3 Place all of the samples on stirring hotplates. Warm and to each client sample or laboratory QA/QC sample add 1 ml of 1.25 M Calcium nitrate solution to each sample and mix thoroughly.

12.11.4 Add 0.2 milliliters of 3.2 M Ammonium phosphate and then add concentrated NH_4OH until a pH of 9.0 or 10.0 is achieved. A fine white precipitate should form.

12.11.5 Allow the precipitate to settle for at least several hours. The precipitate should contain all of the actinides (Thorium, Protactinium, Uranium, etc.) and little if any of the lanthanides or transition metals. Decant the supernatant.

12.11.6 Rinse the supernatant with ten milliliters of deionized water. Vortex mix the sample and then centrifuge to separate the precipitate again. Pour off the su pernatant.

12.11.7 Add five milliliters of concentrated HNO_3 to the sample, transfer the sample to a smaller beaker than the original if necessary and cook the sample down to near dryness. Transfer the sample along with the appropriate paperwork to the separatio ns laboratory.

12.12 Thioacetamide Separation

12.12.1 Transfer the sample to an appropriate sized beaker and place the sample on a stirring hot plate. While the sample is heating, add approximately five grams of potassium metabisulfite to each sample.

12.12.2 Once the sample is near boiling, start adding the 4% Thioacetamide solution in 2 -ml increments. Continue adding the Thioacetamide solution until either a precipitate forms, the sample undergoes a color change, or a total of twelve milliliters of 4% Thioacetamide solution has been added whichever comes first.

12.12.3 While the solution is still hot, filter the sample through a 47 -mm, 0.45mm pore size filter. Dispose of the filter and precipitate and save the filtrate for analysis as per the appropriate section elsewhere in this procedure.

13.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

13.1 All excess sample materials, extracts, byproducts, and associated waste will be disposed of in the appropriate containers and segregated into the appropriate waste streams for final disposal according to the *Waste Management Plan*, and associated documents.

13.2 All laboratory activities associated with this procedure will be carried out in the fashion designed to generate the least amount of waste possible and still achieve the necessary quality of data.

13.3 Pre-cleaned disposable plastic lab ware will be placed in the appropriate waste container following its use in the laboratory.

14.0 CALCULATIONS

The calculations in this procedure are all addition, subtraction, or ratios.

15.0 METHOD PERFORMANCE

There are no quantifiable measurements, which can be made to directly determine the method performance for this procedure. Method performance will be inferred from the performance of laboratory control standards, matrix spikes, and blanks as well as visual observation of the completeness of digestion of samples when total digestion is required.

16.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

16.1 It is the laboratory policy to analyze a Laboratory Control Sample (LCS), a Laboratory Method Blank (MBL), and a Duplicate (DUP) with each work order. Soil samples will be reported on a dry weight basis unless otherwise requested by the client. Work orders are unique for each client, matrix, and isotope.

(The QC measurement acceptance criterion is detailed in each counting procedure).

16.2 RCRA Methods 9310 (Gross Alpha/ Beta); 9315 (Alpha Emitting Radium Isotopes); and 9320 (Radium 228) require a sample duplicate be analyzed at a frequency of one in every ten samples.

17.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

Sample data which is deemed to be unacceptable will be reanalyzed when it is not possible to relate the deficiency to a calculation or clerical error and error and ther e is sufficient sample available for reanalysis.

18.0 REFERENCES

18.1 Standard Methods for the Examination of Water & Wastewater

19.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

19.1 Validation data is available on file.

19.2 There are no tables or diagrams associated with this procedure.

20.0 DEVIATONS FROM REFERENCED PROCEDURES

20.1 The EPA procedures referenced above are not contained in their entirety in this procedure. AP -002 procedure as written only addresses the preliminary chemical preparation step s (digestion, gross precipitation, etc.). The chemical separation steps are contained in separate analysis procedure. Also, though the referenced EPA procedures are only applicable to drinking water samples, the AP - 002 as written contains instructions applicable to other matrices.

20.2 EPA procedure 904.0 for the analysis of Radium-228 in drinking water is subject to interference from stable Barium in the sample. The use of Barium -133 in AP-002 as written eliminates this problem.

20.3 EPA 904.0 procedure for the analysis of Radium-228 in drinking water assumes a 100% recovery for all of the steps included in AP -002. Procedure AP -002 uses a Barium -133 chemical yield tracer to determine an actual chemical recovery as opposed to assuming a recovery.

20.4 Three reagents listed in the EPA procedure 904.0 for Radium -228 are not used in the analysis of Radium-228 by AP-002 and AP-007. Phenolphthalein is used in place of methyl orange, Barium -133 is used in place of the stable Barium carrier, and the Strontium -Yttrium carrier is not needed since the Barium/Lead sulfate carrier is virtually 100% effective for the removal of Radium from water samples. The Actinium is then allowed to ingrow into the separated Radium. This separation of the Barium/Lead Sulfate carrier is less prone to interferences.

20.5 Because of elevated levels of activity, it may be necessary to use aliquots that are smaller than those recommended by the referenced procedures.

Document Revision History

Revision	Effective Date	Changes From Previous Revision
18	10/29/13	· Document Revision History table implemented · Removed reference to obsolete title/position · Changed title *Health and Safety Officer* to *Health and Safety Manager* in Section 6.4 · Corrected formatting inconsistencies throughout document
19	10/29/14	· Corrected spacing format inconsistency in Section 11.2

Eberline Analytical
Oak Ridge Laboratory
Analytical Procedure

AP-005

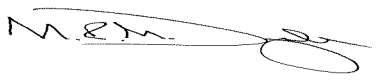
Alpha Isotopic Analyses

AUTHORIZATION AND APPROVAL STATEMENT

This **Eberline Analytical** - Oak Ridge Laboratory, Analytical Procedure,
Alpha Isotopic Analyses
is authorized and approved in its entirety by:

Saba Arnold Seaver
Quality Assurance Manager

Date: October 31, 2014



Michael R. McDougall
Laboratory Manager

Date: October 31, 2014

1.0 SCOPE, PURPOSE, AND APPLICATION

1.1 The purpose of this procedure is to describe chemical separation techniques for the determination of Plutonium, Thorium, and Uranium Alpha -emitting isotopes. Analytical steps include the selective loading and elution of these radionuclides for subsequent micro -precipitation and counting by alpha spectroscopy.

1.2 This procedure does not describe the initial preparation necessary to homogenize and bring samples into the proper conditions of dissolution, acid concentration, and oxidation state appropriate for the chemical separation (Refer to AP -001 and AP-002).

1.3 This procedure is applicable to all sample matrices following the chemical treatment discussed in AP 001 and AP-002. Some deviations may be required for individual samples depending on the matrix and client needs.

2.0 DETECTION LIMITS

2.1 The final determination step for samples prepared using this procedure is counting by alpha spectroscopy using semi-conductor detectors as described in laboratory procedure AP-018. Based on a soil sample of approximately one gram and a water sample of approximately 0.5 liters, the approximate achievable detection limits are as follows:

Solids 1.0 Gram Aliquots	<1.0 pCi/g (See 2.2)
Liquids, 0.5 Liter Aliquots	<1.0 pCi/l (See 2.2)

2.2 These detection limits are based on working knowledge of procedures . Individual sample detection limits may vary from these values based on sample weight/volume , activity present in the sample, potential interferences and counting time.

3.0 SUMMARY OF TEST METHOD

3.1 Thorium-229, Uranium-232, and Plutonium-236 are added as tracers during initial sample preparation and are used to determine chemical recovery. Alpha emitting isotopes are selectively separated as chlorides or nitrates on ion exchange resin columns. Precipitation occurs upon the addition of varying concentrations of hydrofluoric acid, which forms insoluble fluorides that are then filtered through very retentive filter media. The tracer yield and isotopic concentrations or activity are then determined by Alpha spectroscopy.

4.0 DEFINITIONS

4.1 MSDS – Material Safety Data Sheets

4.2 NIST – National Institute of Standards and Technology

4.3 LCS – Laboratory Control Sample

4.4 MS – Matrix Spike

5.0 INTERFERENCES

Samples will demonstrate significant interference if elevated Uranium activity is present in samples. In these instances, special cleanup chemistry is required. Specific steps are included within this procedure for the

cleanup of Uranium. Other interference may include various inorganic constituents. Based on historical or other information of the se types of interferences, special cleanup steps will be provided in written form by the Laboratory Manager for specific analytical cases.

6.0 SAFETY

Laboratory chemical and general safety shall be conducted as required within *Eberline Analytical /Oak Ridge Laboratory, Chemical Hygiene /Health & Safety Plan , Latest Version*

Laboratory radiation safety shall be conducted as required within *Eberline Analytical /Radiation Protection Plan and Attachments , Latest Version*

Waste management and sample return shall be conducted as required within *Eberline Analytical /Waste Management Plan , Latest Version*

6.1 Housekeeping

6.1.1 All work areas shall be kept as clean as possible at all times and the entire work area shall be cleaned at the conclusion of each shift.

6.1.2 Minimize unnecessary items and clutter.

6.1.3 Promptly clean any spills that occur using the guidance contained in the Emergency Action Plan, Spill Response Procedure and support of the Radiation Safety Officer and Health and Safety Officer if necessary.

6.2 Clearly label all sample containers (beakers, bottles, c -tubes etc.) with the work order number, analysis fraction, and analyte identification information such as Total Sr , Iso-U , or some other recognizable wording.

6.3 Any labels that identify the hazards associated with a particular sample container at the time of receipt will remain affixed to that container AND to ALL subsequent sub sampling from, and disposal of, that container.

6.4 Dispose of all waste in the appropriate containers as directed by the Waste Management Pl an.

6.5 Dispose non -rad waste in appropriate containers, DO NOT PUT NON -RAD WASTE INTO RAD WASTE CONTAINERS.

6.6 Personal protective equipment for this procedure shall consist of a lab coat or protective apron, safety glasses or goggles and chemical resistant labo ratory gloves.

7.0 EQUIPMENT AND SUPPLIES

7.1 Beakers, Assorted Sizes/Volumes

7.2 Hot Plate

7.3 Column Support Stands

7.4 Disposable 10-ml Polypropylene Columns, 35 -└rn Porous Polyethylene Frit

7.5 Analytical Balance

7.6 Assorted Tools and Routine Laboratory Equipment

7.7 EiChrom Anion Exchange Resin (chloride form) or equivalent

7.8 Cation Exchange resin, AG 50-X4, 100-200 Mesh

7.9 Membrane Filters, 0.1-µm, 25-mm, HF Resistant

7.10 Whatman Filter Paper, 15-cm or Equivalent

7.11 Membrane Filter, 0.1mm white nitrocellulose, 125mm.

7.12 Membrane filter for carbon suspension (GH-4 0.8 mm)

7.13 The laboratory may use pre-cleaned disposable plastic lab ware as appropriate and applicable to this or any other analytical procedure. Disposable plastic ware will be disposed of in the appropriate waste container after use.

8.0 REAGENTS AND STANDARDS

8.1 Hydrochloric acid, (HCl), concentrated, 12 Normal, reagent grade.

8.2 8N Hydrochloric a cid: Add 667 ml of concentrated HCl to 300 ml of deionized water in a 1 -liter graduated cylinder, mix thoroughly. Add deionized water to reach a total volume of 1 liter and mix again.

8.3 6.5N Hydrochloric acid: Add 542 ml of concentrated HCl to 400 ml of dei onized water in a 1-liter graduated cylinder. Mix thoroughly. Add deionized water to reach a total volume of 1 liter and mix again.

8.4 0.5N Hydrochloric acid: Add 42 ml of concentrated HCl to 900 ml of deionized water in a 1 -liter graduated cylinder. Mix thoroughly. Add deionized water to reach a total volume of 1 liter and mix again.

8.5 Nitric acid, (HNO_3), concentrated, 16 Normal, reagent grade.

8.6 8N Nitric acid: Add 500 ml of concentrated nitric acid to 500 ml of deionized water in a 1 -liter graduated cylinder. Mix thoroughly. Add deionized water to reach a total volume of 1 liter and mix again.

8.7 Hydrofluoric acid, (HF), concentrated, 48%, reagent grade.

8.8 Ammonium iodide, (NH_4I), reagent grade.

8.9 8N HCl -0.1M NH_4I solution: Dissolve 3 grams of Ammonium iodide in 10 0 ml of 8N HCl, mix thoroughly. Add an additional 100 ml of 8N HCl while mixing. Shelf life is one day..

8.10 6.5N HCl -0.04N HF solution: Dilute 3 ml of concentrated HF to 2 -liter volume with 6.5N HCl.

8.11 30% Hydrogen peroxide, (H_2O_2), reagent grade.

8.12 0.5 mg/ml Neodymium (Nd) carrier: commercial Neodymium carrier, spectroscopy grade. (Alternatively, dissolve 1g Nd_2O_3 in 800 milliliters 2% HNO_3. Add 2% HNO_3 to reach a total volume of 1Liter.).

8.13 20% Titanous chloride, reagent grade.

8.14 Carbon substrate preconditioned filter: To 400 ml of deionized water, add 20 ml of concentrated HCl, 10 ml of concentrated HF, 10 mg of Nd carrier. Adjust volume to 500 ml with deionized water and add 5 ml of Carbon suspension. To precondition, filter 10 ml of the above solution through the appropriate filter.

8.15 Cerium Carrier:The laboratory will purchase 10,000 ug/l, (ppm) Atomic Absorption standards. For a 1.0 mg/l solution, dilute 10ml of the 10,000 ug/l to 100ml. Add 1ml of HNO_3, mix very well before use. For a 0.1 mg/l carrier, dilute 10ml of the 1.0mg/l carrier to 100ml, add 1ml of concentrated HNO_3 mix very well before use.

8.16 Carbon suspension. Dissolve 60 GN -4 filters in 50mls of con centrated sulfuric acid and heat to dissolve (black). Then add to 30mls D IH_2O and volume to 100mls total in DiH2O. **CAUTION** be careful in adding sulfuric acid to D IH_2O it will generate significant heat and the boil water.

9.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

9.1 Sample collection and preservation is not the responsibility of the laboratory and is not applicable to this procedure. Upon receipt of water samples, the laboratory may preserve/pH adjust the samples depending on the composition of the sample and the requested analysis.

9.2 Unless otherwise directed by the client, after receipt, all soil, sdid, water, and vegetation samples will be segregated according to preliminary activity scans and stored in a secure, climate controlled location. Tissue samples will be stored in a freezer prior to analysis.

10.0 QUALITY CONTROL

10.1 One Laboratory Control Sample (LCS) shall be analyzed with every 20 samples. The LCS will be prepared and analyzed the same way and along with the analysis batch for the same analytical parameter.

10.2 One analysis blank shall be run with every 20 samples. If there are less than 20 samples per analysis batch, then one blank per batch shall be analyzed.

10.3 A minimum of one or a designated number of client samples shall be duplicated with every 20 samples (one sample for every 10 client samples will be duplicated for RCRA or SW846 analyses). Ifthere are less than 20 samples per analysis batch, then a minimum of one or a sufficient number of duplicates to meet client criteria shall be analyzed per analytical batch. Where the matrix type, limited sample volume or other special considerations preclude this as a viable option, a replicate analysis will be used for QC evaluation.

10.4 If requested by a client, a matrix spike composed of a sample spiked with a standard containing at least one of the isotopes in question (NIST traceable or equivalent) shall be run with each batch.

10.5 Other client specific requirements may supersede the above requirements.

11.0 CALIBRATION AND STANDARDIZATION

11.1 There are no standardized carriers used for this procedure.

11.2 The calibration of the Alpha Spectroscopy detector is covered in procedure(s) AP-018.

11.3 The dilution of NIST traceable (or equivalent) standard solutions is covered in procedure MP -009.

11.4 The calibration verification of the analytical balances is covered in procedure MP -010

11.5 The use, maintenance, and volume verification of the mechanical pipettes is covered in MP-025

12.0 PROCEDURE

All samples should be placed on a hotplate and evaporated to near dryness. Be sure to not let the samples bake into the beakers, but remove all moisture.

12.1 Uranium Analysis

12.1.1 Add three to four milliliters of concentrated HCl to each clean and labeled beaker and heat to dryness to convert the sample to the chloride form.

12.1.2 Prepare a 10 cc disposable ion exchange column with 10 milliliters of EiChrom Anion Exchange Resin (or equivalent) and precondition column with 35 to 40 milliliters of 8N HCl.

12.1.3 Add 20 milliliters of 8N HCl to the sample and warm slightly if necessary to bring the sample into solution. Transfer sample to centrifuge tube with 8N HCl and bring volume up to about 35 ml.

12.1.4 Centrifuge the samples if necessary to remove any insoluble material such as silicates and transfer the solution to the prepared ion exchange column. Wash the centrifuge tube with an additional 20 milliliters of 8N HCl, centrifuge if necessary, and transfer the solution to the ion exchange column. Allow all the liquid to pass through the column.

12.1.5 Wash the column with 35 milliliters of 8N HCl - 0.1N NH_4I, 35 milliliters of 6.5N HCl - 0.04 HF, and ten milliliters of 6.5N HCl. Discard all three washes to the hot sink. These washes remove the Thorium, Plutonium, and Americium/Curium fractions.

12.1.6 Elute the Uranium fraction with 50 milliliters of 0.5N HCl into a clean, dry, labeled 100 -ml beaker. Heat to incipient dryness on a hotplate. Do not allow the samples to spatter or bake.

12.1.7 After the samples have dried, dissolve the sample in 10 milliliters of concentrated HCl and transfer to a 50-milliliter centrifuge tube with deionized water, bring volume to ~15 -ml. Add 0.1 milliliters of Nd carrier and 0.3 milliliters of the 20% Titanous Chlor ide solution.

12.1.8 Add 1 milliliter of concentrated HF to each sample and cool in an ice water bath for at least 60 minutes. Proceed to section 12.4 for filtering instructions.

12.2 Thorium/Actinium Analysis

12.2.1 Add three to four milliliters of concentrated HNO_3 to each beaker and heat to dryness to convert the sample to the nitrate form. Add 20 ml of 8N HNO_3 to sample and warm slightly to bring sample into solution.

12.2.2 Prepare a 10 cc disposable ion exchange column with 10 milliliters of EiChrom Anion exchange resin (or equivalent) and precondition column with 50 milliliters of 8N HNO_3.

12.2.3 Centrifuge the samples if necessary to remove any insoluble material such as silicates and transfer the solution to the prepared ion exchange column. Wash the centrifuge tube with an additional 20 milliliters of 8N HNO_3, centrifuge if necessary, and transfer the solution to the ion exchange column. Allow all the liquid to pass through the column. Repeat with two more 20-milliliter rinses. Discard all rinses and loading solutions

12.2.4 Place a clean, labeled 100-milliliter beaker under the column and elute the Thorium fraction with a 50-milliliter rinse of 8N HCl. Place the sample on a hot plate and evaporate to incipient dryness.

12.2.5 After the samples have dried, dissolve the sample in approximately 10 milliliters of concentrated HCl and transfer to a 50-milliliter centrifuge tube with deionized water, bring volume to ~15-ml. Add 0.75 milliliters of 0.1mg/ml Ce carrier.

12.2.6 Add 1 milliliter of concentrated HF to each sample and cool in an ice water bath f or at least 60 minutes. Proceed to section 12.4 for filtering instructions.

12.3 Plutonium Analysis

12.3.1 Add three to four milliliters of concentrated HNO_3 to each beaker and heat to dryness to convert the sample to the nitrate form. Add 20 ml of 8N HNO_3 to sample and warm slightly to bring sample into solution.

12.3.2 Prepare a 10 cc disposable ion exchange column with 10 milliliters of AG 1 X 8 Anion exchange resin (or equivalent) and precondition column with 50 milliliters of 8N HNO_3.

12.3.3 Transfer the sample to a 50-cc Centrifuge tube with 8N HNO_3 and bring volume to about 35 ml. Centrifuge the samples if necessary to remove any insoluble material such as silicates and transfer the solution to the prepared ion exchange column. Wash the centrifuge tube with an additional 20 milliliters of 8N HNO_3, centrifuge if necessary, and transfer the solution to the ion exchange column. Allow all the liquid to pass through the column. Repeat with two more 20-milliliter rinses of 8N HNO_3. Discard all rinses and loading solutions.

12.3.4 Rinse the column with an additional 35 milliliters of 8N HCl and discard this rinse as well.

12.3.5 Place a clean, labeled 100-milliliter beaker under the column. Elute the Plutonium with 50 milliliters of 8N HCl - 0.1M NH_4I.

12.3.6 Add one milliliter of concentrated HNO_3 to the sample beakers and place on a hotplate to dry to incipient dryness.

12.3.7 After the samples have dried; dissolve the sample in approximately 10 milliliters of concentrated HCl and transfer to a 50-milliliter centrifuge tube with deionized water, bring volume to ~15-ml. Add 0.1 ml Nd carrier and 0.3 ml of the 20% Titanous Chloride solution.

12.3.8 Add 5 milliliters of concentrated HF to each sample and cool in an ice water bath for approximately 60 minutes. Proceed to section 12.4 for filtering instructions.

12.4 Sample Filtering and Mounting

12.4.1 If Plutonium-241 is requested in addition to other Plutonium isotopes, a nitrocellulose filter ***must*** be used for sample mounting:

0.1 mm EiChrom Resolve filter (or equivalent) is used for U, Pu, and Th with carbon substrate

0.1 mm cellulose filter for Pu-241 (no alcohol, no carbon substrate)

12.4.2 Place the filter on top of plastic support in filter apparatus. ***DO NOT*** use carbon substrate or alcohol on the nitrocellulose filter.

12.4.3 Add approximately 5 ml of alcohol to filter. Let set 3 to 5 minutes and check for leaks. Start vacuum pump and let alcohol go through the filter. Add carbon substrate to filter and slowly filter through. Add the sample to the filter, rinse centrifuge tube with 5 -10 ml of deionized water and add to filter.

12.4.4 Rinse the filter with deionized water, and vacuum suction the filter. Discard the filtrate into the hot sink.

12.4.5 Place samples in clean, labeled petri dishes and submit with documentation for counting. Carefully place a ***SMALL*** mark with a pencil or Sharpie marker on the top side of the filter near the edge.

12.5 Plutonium-241 analysis after initial Alpha count: The use of a nitrocellulose in Ultma Gold scintillation cocktail will preclude the need for any ashing or digestion of the filter media. The filter media when exposed to the cocktail becomes optically clear. Do not add water to the filter media prior to adding cocktail, as this will preclude clarification of the filter media. The process blank from Isotopic Plutonium analyses shall be used as the process blank for Plutonium-241 by liquid scintillation.

12.5.1 Transfer the filter to glass liquid scintillation vial.

12.5.2 Mark the vial on the cap and on the bottom of the vial ONLY. Do not place any marks on the sides of the vial.

12.5.3 Add ten milliliters of liquid scintillation cocktail and 10ml of DI Water to blank and samples (Ultima Gold-XR or equivalent). Add 10 mls of scintillation cocktail, the Pu-241 spike as per the LIMS requirement, and 10 mls of DI water to the LCS.

12.5.4 Vigerously shake samples to mix the DI Water with the scintillation cocktail

12.5.5 Transfer the sample along with the associated documentation to the count room for beta liquid scintillation counting.

12.6 Uranium Clean-Up

12.6.1 The chemical preparation lab will have dissolved, digested and concentrated the sample as a calcium phosphate precipitate in a beaker and then converted to a nitrate form. Take the dried sample in the bottom of a beaker and dissolve it using approximately 20 mL of 3N HNO_3 ‼1.0 M $Al(NO_3)_3$.

12.6.2 Add 4 ml of 0.6M Ferrous sulfamate to each sample. Swirl to mix. Wait two or three minutes after mixing.

12.6.3 Add approximately 200 mg of ascorbic acid to each solution and swirl to mix. Wait for two to three minutes.

12.6.4 For each solution, place a UTEVA resin column in the column rack.

12.6.5 Place a beaker below each colum n, remove the bottom plug from each column and allow them to drain. Attach a reservoir to the top of each column.

12.6.6 Place one milliliter (roughly 0.7 grams) of the UTEVA resin in a small diameter ion exchange column and carefully put a plastic frit in plac e on top of the resin.

12.6.7 Pipette 5 mL of 3N HNO_3 into each reservoir to condition the resin. Allow the acid to drain into the beaker. Discard this conditioning solution.

12.6.8 Place a clean, labeled 50 mL c -tube below each column and transfer the dissolved sample to its respective UTEVA column.

12.6.9 Rinse each sample c -tube with 5 mL of 3N HNO_3, add this to the UTEVA column and catch it in the same beaker. Repeat this rinse a second time with another 5 mL portion of 3N Nitric Acid that is also collected in the same beaker. The labeled beaker should now contain Americium, Curium and Plutonium. Barring excessive column loading, the Uranium, Thorium and other isotopes should be retained on the column.

13.0 WASTE MANAGEMENT AND Pollution PREVENTION

13.1 All excess sample mat erials, extracts, byproducts, and associated waste will be disposed of in the appropriate containers and segregated into the appropriate waste streams for final disposal according to the Waste Management Plan, WMP -01.

13.2 All laboratory activities associated w ith this procedure will be carried out in the fashion designed to generate the least amount of waste possible and still achieve the necessary quality of data.

13.3 Pre-cleaned disposable plastic lab ware will be placed in the appropriate waste container following its use in the laboratory.

14.0 CALCULATIONS

After counting the samples by Alpha Spectroscopy, the counting uncertainty and minimum detectable activity (MDA) are calculated in accordance to the equations listed in laboratory procedures AP -018

15.0 METHOD PERFORMANCE

15.1 The initial method performance shall be determined using the method detailed in procedure MP-028.

15.2 The continuing method performance is monitored through the use of the laboratory control standards, blanks and duplicates/replicates.

16.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

It is the laboratory policy to analyze a Laboratory Control Sample (LCS), a Laboratory Method Blank (MBL), and a Duplicate (DUP) with each work order. Soil samples will be reported on a dry weight basis unless otherwise requested by the client. Work orders are unique for ea ch client, matrix, and isotope.

16.1 The data assessment and QC measurement acceptance criteria is detailed in AP -018.

17.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

Sample data that is deemed unacceptable will be reanalyzed when it is not possible to relate the deficiency to a calculation or clerical error and there is sufficient sample available for reanalysis.

18.0 REFERENCES

18.1 "Prescribed Procedures for Measurement of Radioactiv ity in Drinking Water," EPA 600/4/90/032, August 1980.

18.2 Am/Cm in Water and Soil , EML Method Am -01, U.S. Department of Energy, Environmental Measurements Laboratory Procedures Manual (HASL 300), Volume 1, 27th Edition; New York, NY.

18.3 Np/Pu in Water and Soil , EML Method Pu-02, U.S. Department of Energy, Environmental Measurements Laboratory Procedures Manual (HASL 300), Volume 1, 27th Edition; New York, NY.

18.4 Iso-Th in Water and Soil , EML Method Th-01, U.S. Department of Energy, Environmental Measurements Laboratory Procedures Manual (HASL 300), Volume 1, 27th Edition; New York, NY.

18.5 Iso-U in Water and Soil , EML Method U-02, U.S. Department of Energy, Environmental Measurements Laboratory Procedures Manual (HASL 300), Volume 1, 27th Edition; New York, NY.

19.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

19.1 Validation data is available on file.

19.2 There are no tables or diagrams associated with this procedure.

20.0 DEVIATIONS FROM REFERENCED METHODS

20.1 Micro-precipitation is used as the mounting technique for all of the isotopes in this procedure as opposed to electro deposition, which is used for some of the isotopes in the referenced methods.

20.2 The ion exchange resins used in this procedure, EiChrom Anion Exchange Resin (chloride form) (or equivalent), are equivalent in function or superior to the resins described in the referenced procedures.

Document Revision History

Revision	Effective Date	Changes From Previous Revision
17	10/31/13	· Document Revision History table implemented · Updated brand of Anion Exchange Resin used throughout procedure · Reworded step 12.2.1 for clarity · Added leak check requirement to step 12.4.3 · Reworded Section 12.5 for clarity · Changed paperwork to documentation throughout procedure
18	10/31/14	· Added Section 20.0 DEVIATIONS FROM REFERENCED METHODS to comply with analytical procedure section requirements set forth in Eberline procedure MP-004 *Preparation and Modification of Procedures and Guidance Documents* · Moved method deviation information from Section 3.0 SUMMARY OF TEST METHOD to Section 20.0

Eberline Analytical
Oak Ridge Laboratory
Analytical Procedure

AP-006

Radium-226 Analysis

AUTHORIZATION AND APPROVAL STATEMENT

This **Eberline Analytical** - Oak Ridge Laboratory, Analytical Procedure,
Radium-226 Analysis
is authorized and approved in its entirety by:

Saba Arnold Seaver
Quality Assurance Manager

Date: October 31, 2014

Michael R. McDougall
Laboratory Manager

Date: October 31, 2014

1.0 PURPOSE, SCOPE, AND APPLICATION

1.1 The purpose of this procedure is to provide detailed instruction for the determination for Radium-226 by Alpha Spectroscopy. This procedure provides detailed instructions for the analysis of water, soil, solids, air filters and other matrices.

1.2 The starting point for this procedure is AP-002 Section 12.1 for water and 12.2 for solid matrices. The scope of this procedure is limited to the final chemical separation of Radium isotopes and the procedure continues to the final sample mount prior to submission for Alpha counting (AP-018).

1.3 This procedure is applicable to all sample matrices though deviations may be required for individual samples.

2.0 DETECTION LIMITS

2.1 Samples for this procedure are counted using an alpha spectroscopy system with semiconductor diode detectors. The anticipated detection limits for this procedure are as follows:

Solids:	1.0 gram	180 min count time	<1.0 picoCuries per gram
Liquids:	0.5 liter	180 min count time	<1.0 picoCuries per liter

2.2 These detection limits are based on working knowledge of the procedure. Individual sample detection limits may vary based on sample matrix, aliquot size, potential interferences and the sample counttime.

3.0 SUMMARY OF TEST METHOD

Radium isotopes are initially separated as precipitates of Radium, Barium and Lead sulfates, either directly from water samples or after mixed acid digestions for solids. Barium-133 is added as analytical tracer for determination of chemical yield. As part of the initial preparation, the sample pH is adjusted to 2.8-3.0 prior to $PbSO_4$ & $BaSO_4$ precipitations. Precipitation occurs upon the addition of ammonium or potassium sulfate, which provides the sulfate ions necessary for this reaction. Precipitates are dissolved in alkaline EDTA, and Radium-226 is selectively reprecipitated as Radium/Barium sulfate by the addition of Acetic acid to slighlty lower the pH. Lead and other interfering compounds remain soluble in mildly acidic EDTA complexes, and are therefore not present in the final precipitate. Radium-226 is prepared for counting by co-precipitation with Barium sulfate, filtered on a 25 mm 0.45 µm filter and then counted by alpha spectroscopy. Barium-133 recovery (used in the determination of chemical yield) is determined by HPGe gamma spectroscopy analysis.

4.0 DEFINITIONS

4.1 MSDS – Material Safety Data Sheets

4.2 NIST – National Institute of Standards and Technology

4.3 EDTA – Ethylenediamine Tetracetic Acid

4.4 TSS – Total Suspended Solids

4.5 TDS – Total Dissolved Solids

4.6 LIMS – Laboratory Information Management System

5.0 INTERFERENCES

Solid or liquid samples that contain significant amounts of elemental Barium are not amenable to this procedure. Elemental Barium will be co-precipitated with the Radium and the excessive mass will interfere with the spectral resolution of alpha particles emitted from Ra-226.

6.0 SAFETY

Laboratory chemical and general safety shall be conducted as required within *Eberline Analytical /Oak Ridge Laboratory, Chemical Hygiene /Health & Safety Plan , Latest Version*

Laboratory radiation safety shall be conducted as required within *Eberline Analytical /Radiation Protection Plan and Attachments , Latest Version*

Waste management and sample return shall be conducted as required within *Eberline Analytical /Waste Management Plan , Latest Version*

6.1 Housekeeping

6.1.1 All work areas shall be kept as clean as possible at all times and the entire work area shall be cleaned at the conclusion of each shift.

6.1.2 Minimize unnecessary items and clutter in the work space.

6.1.3 Promptly clean any spills that occur using the guidance contained in the Emergency Action Plan, Spill Response Procedure and support of the Radiation Safety Officer and Health and Safety Manager if necessary.

6.2 Clearly label all sample containers (beakers, bottles , c-tubes etc.) with the work order number, analysis fraction, and analyte identification information such as Total Sr , Iso-U , or some other recognizable wording.

6.3 Any labels that identify the hazards associated with aparticular sample container at the time of receipt will remain affixed to that container AND to ALL subsequent sub sampling from, and disposal of, that container.

6.4 Dispose of all waste in the appropriate containers as directed by the Waste Management Plan.

6.5 Dispose non -rad waste in appropr iate containers, DO NOT PUT NON -RAD WASTE INTO RAD WASTE CONTAINERS.

6.6 Personal protective equipment for this procedure shall consist of a lab coat or protective apron, safety glasses or goggles and chemical resistant laboratory gloves.

7.0 EQUIPMENT AND SUPPLIES

7.1 Magnetic stir-hot plat

7.2 Alpha spectrometer and Gamma spectrometer

7.3 Vortex stirrer

7.4 Membrane filtering apparatus

7.5 10-ml disposable pipettes

7.6 Transfer pipettes

7.7 Magnetic stir bars

7.8 50-ml centrifuge tubes

7.9 Tuffryn membrane filters (HT -200, 25 mm, 0.45 µm)

7.10 Syringe filters (HT Tuffryn, 25 mm, 0.45 µm)

7.11 Plastic disposable syringe, (20 cc)

7.12 Analytical balance

7.13 Petri dishes, 50 x 9 mm

7.14 The laboratory may use pre-cleaned disposable plastic lab ware as appropriate and applicable to this or any other analytical procedure. Disposable plastic ware will be disposed of in the appropriate waste container after use.

8.0 REAGENTS AND STANDARDS

8.1 Acetic acid, (CH_3COOH), concentrated, glacial, 17.4 Normal, reagent grade

8.2 Ammonium hydroxide, (NH_4OH), concentrated, 15 Normal, reagent grade

8.3 Ammonium sulfate, 200 mg/ml: Dissolve 200 grams of (NH4)2SO4 in deionized water and dilute to 1000 ml in a graduated cylinder with deionized water.

8.4 Alkaline EDTA, 0.25M (Ethylenediamine Tetracetic Acid): Dissolve 20 grams of Sodium hydroxide in 800 ml of deionized water, then slowly add 93 grams of EDTA. Stir and dilute to 1 liter with deionized water in a graduate cylinder. Add drops of phenolphthalein per liter to achieve pink color and demonstrate that the solution is alkaline.

8.5 Nitric Acid, (HNO_3), concentrated, 16 Normal, reagent grade.

8.6 Phenolphthalein: Dissolve 0.1 g of phenolphthalein in 60 mls of reagent alcohol and dilute to 100 mls with deionized water.

9.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

9.1 Sample collection and preservation is not the responsi bility of the laboratory and is not applicable to this procedure. Upon receipt of water samples, the laboratory may preserve/pH adjust the samples depending on the composition of the sample and the requested analysis.

9.2 Unless otherwise directed by the client, after receipt, all soil, solid, water, and vegetation samples will be segregated according to preliminary activity scans and stored in a secure, climate controlled location. Tissue samples will be stored in a freezer prior to analysis.

10.0 QUALITY CONTROL

10.1 One Laboratory Control Sample (LCS) shall be analyzed with every 20 samples. The LCS will be prepared and analyzed the same way and along with the analysis batch for the same analytical parameter.

10.2 One analysis blank shall be run with every 20 samples. If there are less than 20 samples per analysis batch, then one blank per batch shall be analyzed.

10.3 A minimum of one or a designated number of client samples shall be duplicated with every 20 samples (one sample for every 10 client samples will be duplicated f or RCRA or SW846 analyses). If there are less than 20 samples per analysis batch, then a minimum of one or a sufficient number of duplicates to meet client criteria shall be analyzed per analytical batch. Where the matrix type, limited sample volume or oth er special considerations preclude this as a viable option, a replicate analysis will be used for QC evaluation.

10.4 If requested by a client, a matrix spike composed of a sample spiked with a standard containing at least one of the isotopes in question (NIST traceable or equivalent) shall be run with each batch.

10.5 Other client specific requirements may supersede the above requirements.

11.0 CALIBRATION AND STANDARDIZATION

11.1 There are no standardized carriers for this procedure.

11.2 The calibration of the alpha spectroscopy detector is covered in procedure(s) AP-018.

11.3 The dilution of NIST traceable (or equivalent) standard solutions is covered in procedure MP -009.

11.4 The calibration verification of the analytical balances is covered in procedure MP -010

11.5 The use, maintenance, and volume verification of the mechanical pipettes is covered in MP-025

12.0 PROCEDURE

Sign the internal chain of custody forms upon receipt of samples from sample preparation. Samples as prepared are lead and barium sulfates contained within a 50 ml centrifuge tub e.

12.1 Add 1 to 2 drops of phenolphthalein.

12.2 Dissolve the precipitate by adding 20 ml of alkaline EDTA. Heat and vortex the sample if necessary to assist dissolution of lead and barium sulfate precipitates. The pH should be 8.2 -10.00 (pink). If necessary, use concentrated Ammonium hydroxide to adjust pH up. Color range for phenolphthalein is colorless (pH 0-8.1) and pink (pH 8.2-10.00).ntrifuge and syringe filter through a 0.45µm filtersamples if necessary and transfer the supernatant to a clean, labele d centrifuge tube. Add 4 ml of 200 mg/ml Ammonium sulfate and mix.

12.3 Add 2ml of Acetic acid, and gently swirl until a precipitate forms. acid.

NOTE

If Ra-228 is required, record the date and time within the LIMS, Laboratory Technician Functions and record the first separation time as t0 , as the start of the ingrowth of Ac-228.

12.6 Filter the precipitate with vacuum onto a tarred 0.45 µm Tuffryn membrane filter.

12.7 Rinse the centrifuge tube with deionized water into the filtering funnel, and rinse the filtering funnel with deionized water. Discard the filtrate to the Satellite Accumulation Area (SAA) Pb/EDTA waste stream receptacle.

12.7 Remove filter, and dry then place in a 50 x 9 mm petri dish. Obtain final weight of filter. Document tare and final filter weights within the LIMS section, Laboratory Technician Functions, Gravimetric/Count Wt. , print Gravimetric Weight data sheet and place in analytical sub -file.

12.8 Print the analysis sheet from the LIMS within, Laboratory Technician Functions, Print Analysis Sheet and place in analytical sub-file. Complete internal chain of custody information and transfer samples to the count room.

13.0 WASTE MANAGEMENT AND POLUTION PREVENTION

13.1 The filtrate for 12.7 above is collected in the Pb/EDTA Waste receptacle in the laboratory before being moved to the waste accumulation area.

13.2 All excess sample materials, extracts, byproducts, and associated waste will be disposed of in the appropriate containers and segregated into the appropriate waste streams for final disposal according to the Waste Management Plan, WMP-01.

13.3 All laboratory activities associated with this procedure will be carried out in the fashion designed to generate the least amount of waste possible and still achieve the necessary quality of data.

13.4 Pre-cleaned disposable plastic lab ware will be placed in the appropriate waste container following its use in the laboratory.

14.0 CALCULATIONS

After counting the samples by Alpha Spectroscopy, the counting uncertainty and minimum detectable activity (MDA) are calculated in accordance to the equations listed in the laboratory procedure AP-018.

15.0 METHOD PERFORMANCE

15.1 The initial method performance shall be determined using the method detailed in procedure MP -028.

15.2 The method performance is continuously monitored using the laborato ry control standards, blanks and duplicates/replicates.

16.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY ASSURANCE MEASUREMENTS

It is the laboratory policy to analyze a Laboratory Control Sample (LCS), a Laboratory Method Blank (MBL), and a Duplicate (DUP) with each work order. Soil samples will be reported on a dry weight basis unless otherwise requested by the client. Work orders are unique for each client, matrix, and isotope. Specific client requirements may supersede the following laboratory default criteria.

16.1 The data assessment and QC measurement acceptance criteria is detailed in AP_018.

16.2 RCRA Methods 9310 (Gross Alpha/ Beta); 9315 (Alpha Emitting Radium Isotopes); and 9320 (Radium 228) require a sample duplicate be analyzed at a frequency of one i n every ten samples.

17.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

Sample data that is deemed to be unacceptable will be reanalyzed when it is not possible to relate the deficiency to a calculation or clerical error and there is sufficient sample available for reanalysis.

18.0 REFERENCES

18.1 "Handbook for Analytical Quality Control in Radioanalytical Laboratories", EPA-600/7-77-088, August 1977.

18.2 "Standard Methods for the Examination of Water and Waste Water" APHA-AWWA-WPCF, 2005, 21st Edition.

18.3 Ra-226 in Water and Soil (Chemical Prep) , EPA Method 903.0, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory Procedures Manual (EPA-600/4-80-032, 8/80) Cincinnati, OH.

18.4 Ra-226 in Soil (Gamma) , LANL Method ER -130, Los Alamos National Laboratory Procedures Manual, Los Alamos, NM.

19.0 TABLES, DIAGRAMS, AND VALIDATION DATA

19.1 There are no tables or diagrams associated with this procedure.

19.2 Validation data is available on file.

20.0 DEVIATIONS FROM REFERENCED PROCEDURES

The EPA method 903.0 uses Sodium hydroxide for the adjustment of pH of the EDTA solution. This procedure as written uses Ammonium hydroxide. The chemical action of these two bases in the same without the creation of an additional cation (Na).

Document Revision History

Revision	Effective Date	Changes From Previous Revision
14	10/31/13	· Document Revision History table implemented · Added LIMS to list of definitions in Section 4.0 · Changed title *Health and Safety Officer* to *Health and Safety Manager* in Section 6.1.3
15	10/31/14	· Reviewed: no changes necessary

Eberline Analytical
Oak Ridge Laboratory
Analytical Procedure

AP-007

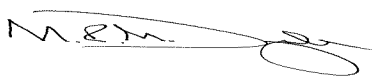
Radium-228 Analysis

AUTHORIZATION AND APPROVAL STATEMENT

This **Eberline Analytical** - Oak Ridge Laboratory, Analytical Procedure,
Radium-228 Analysis
is authorized and approved in its entirety by:

Saba Arnold Seaver
Quality Assurance Manager

Date: October 31, 2014



Michael R. McDougall
Laboratory Manager

Date: October 31, 2014

1.0 SCOPE, PURPOSE, AND APPLICABLE MATRICES

1.1 The purpose of this procedure is to provide detailed instruction for the determination for Radium-228 by Gas Proportional Counting.

1.2 This procedure assumes that the sample is presented as a sulfate precipitate (AP-002, steps 12.1, 12.2, and 12.3) and covers the chemical separation of the Actinium-228 daughter from the Radium-228 parent.

1.3 This procedure is applicable to all sample matrices though deviations may be required for individual samples.

1.4 This procedure is applicable to multiple matrices (as can be prepared using AP-002), and the measurement of Radium-228 in drinking water. The Interim Primary Drinking water regulations state if the alpha screening test reveals a gross alpha activity above 5 pCi/l, a Radium-226 analysis must also be performed. If the level of Radium-226 is above 3 pCi/l, the sample must also be measured for Radium-228.

2.0 DETECTION LIMITS

2.1 The samples for this procedure are counted using a beta gas flow proportional detector. The anticipated detection limits for this procedure are as follows:

Solids:	1.0 g sample,	120 min count time,	<2.0 pCi/g
Liquids:	1.0 l sample,	120 min. count time,	<2.0 pCi/l

2.2 These detection limits are based on technical experience. Individual sample detection limits may vary.

3.0 SUMMARY OF TEST METHOD

Radium-228 in samples is separated as a precipitate of the sulfates of Radium, Barium and Lead. In successive precipitations, Lead and other interferences are removed by selective precipitation of Radium from slightly acidic EDTA. Further Lead removal is achieved by precipitation as Lead sulfide. After a sufficient ingrowth period, Actinium-228 is separated from Radium-228 by multiple precipitations as Yttrium hydroxide. Actinium-228/Yttrium oxalate, precipitate is filtered and then counted on gas proportional detectors. Radium-228 activity is calculated from Beta activity present in this final precipitate.

4.0 DEFINITIONS

4.1 MSDS – Material Safety Data Sheets

4.2 NIST – National Institute of Standards and Technology

4.3 EDTA – Ethylenediamine Tetracetic Acid

4.4 TSS – Total Suspended Solids

4.5 TDS – Total Dissolved Solids

4.6 GPC – Gas Proportional Counter

4.7 LIMS – Laboratory Information Management System

5.0 INTERFERENCES

5.1 Yttrium-90 is not completely separated from Ac-228. Samples that are known to contain Strontium-90 cannot be accurately analyzed by this method without significant modification. In the event that a sample is know n to contain Strontium/Yttrium -90 activity, these samples shall be analyzed by the EiChroM method.

5.2 Since Barium-133 and stable Yttrium are used as carriers for the procedure as written, natural Barium in the sample will NOT interfere in the analysis of Radium-228; however, will cause incorrect Barium-133 yield determinations. In these cases the gravimetric Yttrium recovery shall only be used for yield determinations.

6.0 SAFETY

Laboratory chemical and general safety shall be conducted as required within *Eberline Analytical /Oak Ridge Laboratory, Chemical Hygiene /Health & Safety Plan , Latest Version*

Laboratory radiation safety shall be conducted as required within *Eberline Analytical /Radiation Protection Plan and Attachments , Latest Version*

Waste management and sample return shall be conducted as required within *Eberline Analytical /Waste Management Plan , Latest Version*

6.1 Housekeeping

6.1.1 All work areas shall be kept as clean as possible at all times and the entire work area shall be cleaned at the conclusion of each shift.

6.1.2 Minimize unnecessary items and clutter in the work space.

6.1.3 Promptly clean any spills that occur using the guidance contained in the Emergency Action Plan, Spill Response Procedure and support of the Radiation Safety Officer and Health and Safety Manager if necessary.

6.2 Clearly label all sample containers (beakers, bottles, c-tubes etc.) with the work order number, analysis fraction, and analyte identification information such as Total Sr , Iso-U , or some other recognizable wording.

6.3 Any labels that identify the hazards associated with a particular sample container at thetime of receipt will remain affixed to that container AND to ALL subsequent sub sampling from, and disposal of, that container.

6.4 Dispose of all waste in the appropriate containers as directed by the Waste Management Plan.

6.5 Dispose non -rad waste in appropriat e containers, DO NOT PUT NON -RAD WASTE INTO RAD WASTE CONTAINERS.

6.6 Personal protective equipment for this procedure shall consist of a lab coat or protective apron, safety glasses or goggles and chemical resistant laboratory gloves.

7.0 EQUIPMENT AND SUPPLIES

7.1 Assorted glassware

7.2 Transfer pipettes

7.3 Magnetic stirrer/hot plate and magnetic stir bars

7.4 50-ml centrifuge tubes

7.5 Centrifuge

7.6 Vortex stirrer

7.7 Tuffryn 47mm, 0.2 µm filter paper, (Pall 66199)

7.8 Gross Alpha/Beta GPC counter

7.9 Air displacement pipette: 50, 100, 300-µL and 1-ml

7.10 Liquid scintillation vials (23-ml capacity)

7.11 0.47mm filter apparatus

7.12 The laboratory may use pre-cleaned disposable plastic lab ware as appropriate and applicable to this or any other analytical procedure. Disposable plastic ware will be disposed ofin the appropriate waste container after use.

8.0 REAGENTS AND STANDARDS

8.1 5% Ammonium oxalate: Dissolve 50grams of $(NH_4)_2C_2O_4 \cdot H_2O$ in deionized water and dilute to 1000ml.

8.2 2% Ammonium sulfide: Dilute 5 ml of 20 -24% $(NH_4)_2S$ to 50 ml with deionized water. Due to the tendency of this reagent to disassociate and evolve Hydrogen sulfide, this solution needs to be prepared weekly.

8.3 Alkaline EDTA, 0.25M: Dissolve 20 grams NaOH in 750 ml water, heat and slowly add 93 grams of EDTAwhile stirring. After the salt is dissolved, dilute volume to one liter. Add drops of phenolphthalein to achieve pink color and demonstrate that the solution is alkaline.

8.4 1.5 mg/ml Lead carrier: Dissolve 2.397 grams of $Pb(NO_3)_2$ in deionized water and volume to 1000 ml with deionized water.

8.5 Nitric acid, (HNO_3), 16N, 70%, reagent grade

8.6 6N Nitric acid: Add 375 ml of 16N, 70% Nitric acid to 500 ml of deionized water in a 1-liter graduated cylinder. Dilute to volume after it has cooled to room temperature.

8.7 1N Nitric acid: Add 63 ml of 16N, 70% Nitr ic acid to 500 ml deionized water in a 1 -liter graduated cylinder, and dilute to volume after it has reached room temperature.

8.8 Phenolphthalein: Dissolve 0.1 g of phenolphthalein in 60 mls of reagent alcohol and dilute to 100 mls with deionized water.

8.9 18N Sodium hydroxide: Slowly dissolve 720 grams of NaOH pellets in deionized water, and dilute to 1000 ml after it has reached room temperature.

8.10 10N Sodium hydroxide: Slowly dissolve 400grams of NaOH pellets in deionized water, and dilute to 1000ml when it has reached room temperature.

8.11 9 mg/ml Yttrium carrier: Dissolve 11.425 grams Y_2O_3 in an Erlenmeyer flask containing 20 ml of deionized water. Heat to boiling and continue stirring on a magnetic stirrer/hot plate while adding concentrated HNO_3 in small amou nts. Usually about15ml of Nitric acid is necessary to complete dissolution. Small amounts of deionized water may be required to replace that lost by evaporation. After total dissolution, add 35ml of concentrated Nitric acid and dilute to 1 liter with deio nized water. This solution should be standardized as detailed in section 11.0.

9.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

9.1 Sample collection and preservation is not the responsibility of the laboratory and is not applicable to this procedure. Upon receipt of water samples, the laboratory may preserve/pH adjust the samples depending on the composition of the sample and the requested analysis.

9.2 Unless otherwise directed by the client, after receipt, all soil, solid, water, and vegetation samples will be segreg ated according to preliminary activity scans and stored in a secure, climate controlled location. Tissue samples will be stored in a freezer prior to analysis.

10.0 QUALITY CONTROL

10.1 One Laboratory Control Sample (LCS) shall be analyzed with every 20 samples. T he LCS will be prepared and analyzed the same way and along with the analysis batch for the same analytical parameter.

10.2 One analysis blank shall be run with every 20 samples. If there are less than 20 samples per analysis batch, then one blank per batch sh all be analyzed.

10.3 A minimum of one or a designated number of client samples shall be duplicated with every 20 samples (one sample for every 10 client samples will be duplicated for RCRA or SW846 analyses). If there are less than 20 samples per analysis batc h, then a minimum of one or a sufficient number of duplicates to meet client criteria shall be analyzed per analytical batch. Where the matrix type, limited sample volume or other special considerations preclude this as a viable option, a replicate analysis will be used for QC evaluation.

10.4 If requested by a client, a matrix spike composed of a sample spiked with a standard containing at least one of the isotopes in question (NIST traceable or equivalent) shall be run with each batch.

10.5 Other client specific requirements may supersede the above requirements.

11.0 CALIBRATION AND STANDARDIZATION

11.1 Yttrium Carrier standardization, run a minimum of five Carrier samples and determine statistically valid average. Standardization shall be conducted using the same method as chemistry during the analysis process.

11.1.1 Add 2ml of new Yttrium carrier to a 50ml centrifuge tube. Add 20ml of alkaline EDTA and swirl. Add 10ml of 18M NaOH and swirl and place into a hot water bath for 15 minutes. Centrifuge and discard the EDTA. Add 2 ml 6M HNO_3, 5ml DI H_2O and then 3ml of 10M NaOH. Place into a hot water bath for 15 minutes and centrifuge. Discard supernate. Add ~2ml of 1M HNO_3 5ml of DI water and 4ml of 5% Ammonium Oxalate. Filter precipitates on pre-tarred Tuffryn 0.2 µm, 47mm fi lters. Wash filtrate one time with DI Water. Remove filters and, heat lamp dry and reweigh. Standardize using net equivalent mass/ml.

12.0 PROCEDURE

12.1 If the sample is to be analyzed for Radium -228 following Ra-226 determination, the sample for this analysis will normally be contained on a 25-mm filter.

12.2 If the sample is intended only for Radium -228, it will be received as a sulfate precipitate in a 50 -CC centrifuge tube. Follow procedure AP-006, steps 12.1 through 12.7. Remove filter, dry and submit to the count room for Barium-133 count rate determination. Proceed to 12.3.

12.3 Record the initial Barium/Radium sulfate precipitation time as T0 for all Ra -228 analysis in the LIMS Laboratory Technician Functions, Record t0.

12.4 Place the filter into 50ml centrifuge tube containing 20 ml of 0.25M EDTA or add 20 ml of the EDTA solution to the centrifuge tube containing the sulfate precipitate.

12.5 The pH of samples at this step is very important. After dissolution of the sample in 20ml EDTA (preferably an overnight soaking in 20 ml of EDTA), adjust the pH of the solution to between 7.0 and 8.0 using exactly 18 drops of concentrated Nitric acid. Add 2ml of 9 -mg/ml Yttrium carrier.

12.6 Add 0.25ml of 2% $(NH4)_2S$ and stir well. Add 2ml of 1.5 mg/ml lead carrier. .Add 20 drops of 1 0m NaOH, vortex and centrifuge. Centrifuge and pour the supernatant into a clean centrifuge tube. Discard precipitate to lead waste by rinsing c-tube with a minimal amount of water.

NOTE

Due to the short decay time of Actinium-228, the procedure must be carried through to completion after beginning step 12.8

12.7 To the supernatant add, add 0.25 ml of 2% Ammonium sulfide 2 ml of 1.5 mg/ml Lead carrier and, if necessary, add a few drops of 10N NaOH to precipitate PbS, (Lead Sulfide), centrifuge and syringe filter the supernatant into a new centrifuge tube to remove all of the precipitate. Discard the precipitated lead. If necessary this can be a stopping point in the procedure.

12.8 To the supernatant, add 10ml of 18N NaOH, record t1, stir well and heat in a hot wat er bath for 15 minutes, (a visible precipitation should be present). Centrifuge and decant the supernatant to the lead waste container. This is the end of the Actinium-228 ingrowth time and the beginning of Actinium-228 decay.

12.9 Dissolve the precipitate in 2 ml of 6N HNO_3.

12.10 Add 5 ml deionized water, stir and add 3 ml of 10N NaOH to precipitate Yttrium hydroxide. Stir and heat in a hot water bath until the precipitate coagulates. Centrifuge and discard the supernatant to the hot sink.

12.11 Dissolve the precipitate with ~2 ml 1N HNO_3.

12.12 Add 4 ml 5% Ammonium oxalate. Slurry the precipitate with 5ml DI H_2O and vortexing. Then filter through a tarred 47mm 0.2um filter.

12.13 Dry the filter under a heat lamp and re-weigh to obtain recovery.

12.14 Mount the filter on a planchet and cover with aluminum foil.

12.15 Print the analysis sheet from the LIMS within, Laboratory Technician Functions, Print Analysis Sheet and place in analytical sub-file. Complete internal chain of custody information and transfer samples to count room.

13.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

13.1 All excess sample materials, extracts, byproducts, and associated waste will be disposed of in the appropriate containers and segregated into the appropriate waste streams for final disposal according to the Waste Management Plan, WMP-01.

13.2 All laboratory activities associated with this procedure will be carried out in the fashion designed to generate the least amount of waste possible and still achieve the necessary quality of data.

13.3 Pre-cleaned disposable plastic lab ware will be placed in the appropriate waste container following its use in the laboratory.

14.0 CALCULATIONS

After counting the samples by Gas Proportional Counting, the counting uncertainty and minimum detectable activity (MDA) are calculated in accordance to the equ ations listed in laboratory procedure AP-029.

15.0 METHOD PERFORMANCE

15.1 The initial method performance shall be determined using the method detailed in procedure MP-028.

15.2 The method performance is continuously monitored using the laboratory control standards, blanks and duplicates/replicates.

16.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

It is the laboratory policy to analyze a Laboratory Control Sample (LCS), a Laboratory Method Blank (MBL), and a Duplicate (DUP) with each work order. Soil samples will be reported on a dry weight basis unless otherwise requested by the client. Work orders are unique for each client, matrix, and isotope.

16.1 The data assessment and QC measurement acceptance criteria is detailed in AP_029.

16.2 RCRA Methods 9310 (Gross Alpha/ Beta); 9315 (Alpha Emitting Radium Isotopes); and 9320 (Radium 228) require a sample duplicate be analyzed at a frequency of one in every ten samples.

17.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

Sample data which is deemed to be unacceptable will be reanalyzed when it is not possible to relate the deficiency to a calculation or clerical error and there is sufficient sample available for reanalysis.

18.0 REFER1ENCES

18.1 *Ra-228 in Drinking Water (Chemical Prep)* , EPA Method 904.0, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory Procedures Manual (EPA-600/4-80-032, 8/80) Cincinnati, OH.

19.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

19.1 Validation data is available file.

19.2 There are no tables or diagrams associated with this procedure.

20.0 DEVIATIONS FROM REFERENCED METHODS

NOTE

All deviations from EPA procedures apply to both EPA 904.0 and SW -846 9320

20.1 The stable Barium carrier, and the Strontium -Yttrium carrier is not needed since the Barium/Lead sulfate carrier is known to be virtually 100% effective for the removal of Radium from water samples and the Actinium is then allowed to ingrow into the separated Radium. This separation is less prone to interferences than the EPA method, which uses additional carri ers.

20.2 Step 8.12 of the EPA procedure uses 1ml of the 9mg/ml Yttrium carrier and 1ml of the 1.5 mg/ml lead carrier. . These amounts are doubled in step 12.4 of this procedure to provide a more effective chemical separation in all samples.

20.3 The EPA procedure uses 0.3 milliliters of the 2% Ammonium sulfide solution while this procedure as written uses 0.25 milliliters. This difference is not significant.

20.4 The EPA procedure requires stirring the sulfide precipitation reaction (step 8.13) intermittently for about 10 minutes. This procedure requires two 30-second mixing sessions. We have found through experience that this is sufficient.

20.5 In step 8.14 of the EPA procedure, the lead sulfide precipitation is carried out using 1 ml of lead carrier and 0.1 ml of the ammonium sulfide solution. This procedure uses 1.5 ml of lead carrier and 0.25 milliliters of lead sulfide to enhance the separation of this step.

20.6 In step 8.15 of the EPA procedure, 5 milliliters of 18N NaOH is added to the sample. This procedure uses 10 m illiliters of the NaOH solution. This ensures a more complete precipitation and does not adversely affect the analysis.

Document Revision History

Revision	Effective Date	Changes From Previous Revision
19	10/31/13	· Document Revision History table implemented · Added LIMS to list of definitions in Section 4.0 · Changed title *Health and Safety Officer* to *Health and Safety Manager* in Section 6.1.3
20	10/31/14	· Reviewed: no changes necessary